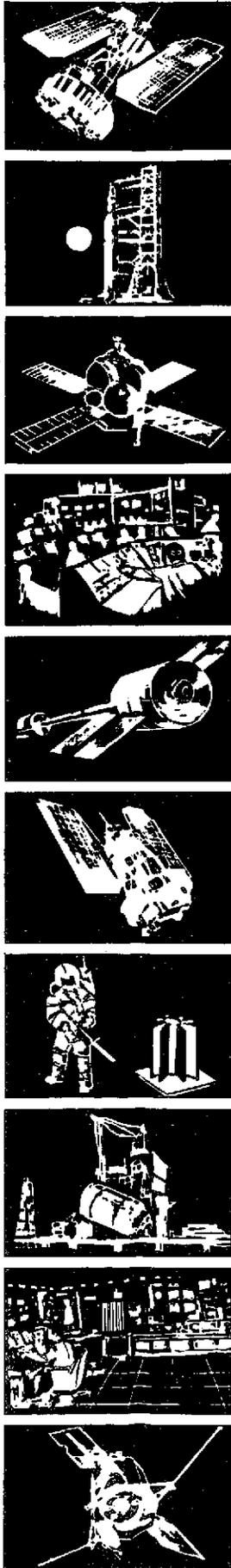


**SPACE
DIVISION**

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**STUDY FOR
 IDENTIFICATION OF
 BENEFICIAL
 USES OF
 SPACE
 (PHASE II)**



**FINAL REPORT
 VOLUME II, BOOK 2
 TECHNICAL REPORT - APPENDICES**

**CONTRACT NAS8-28179
 NOVEMBER 1, 1973
 SUBMITTED PER DPD #296, DR #MA-04**

GENERAL  ELECTRIC

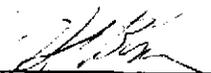
STUDY FOR
IDENTIFICATION OF
BENEFICIAL USES OF SPACE (B.U.S.)
(PHASE II)

CONTRACT NAS8-28179

FINAL REPORT - VOLUME II, BOOK 2
TECHNICAL REPORT - APPENDICES

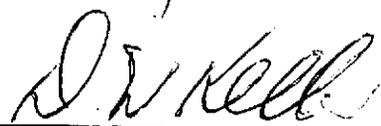
November 1, 1973

Submitted Per DPD #296, DR #MA-04


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SPACE DIVISION

GENERAL  ELECTRIC

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PREFACE

This Final Report on Phase II of the Study for Identification of Beneficial Uses of Space (B.U.S.) is comprised of two volumes:

Volume I - Executive Summary

Volume II - Technical Report

Volume II is further subdivided:

Book 1 - Sections I through IV, Introduction, Method of Study, Study Results, Conclusions and Recommendations

Book 2 - Section V, Appendices A through D

General Electric's Space Division, under contract from the NASA's Marshall Space Flight Center completed Phase I of the Study in December, 1972, and a Final Report for that phase was issued shortly thereafter.

In Phase II, conducted from December, 1972 to December, 1973, the Study has progressed to the investigation of the technology and programmatic involved in development of four of the products selected from those identified by User organizations who participated in Phase I. The selected Products/Organizations were:

- o Surface Acoustic Wave Components.....GE, Electronics Laboratory
- o Transparent Oxides.....Corning Glass Works
- o High Purity Tungsten X-Ray Targets...GE, Medical Systems, Business Division
- o High Specificity Isoenzymes.....Polysciences, Inc.

The methodology employed in this investigation and the results of that effort are reported herein.

The participating organizations supported GE Space Division efforts by evaluating more than 30 major alternative candidate approaches for producing the above products, selecting specific processes for further study, and identifying requirements for nearly 70 experiment and test series necessary to the development of the selected processes.

Subsequent assembly of preliminary program timelines and milestones for such developments revealed a "comfortable" schedule of analyses, experiments and tests, design, development testing and fabrication of operational equipment; with reasonable tolerances in timing to allow for early redesigns, major retests, re-evaluation of results, and moderate redirection; and culminating in 1982-1983 operational dates.

In addition to those Key Individuals from the participating User organizations who contributed specific product, process and planning data in each of their respective areas, the Study Manager acknowledges the considerable contributions of Mr. U. Alvarado and Mr. M. Clarke of the Study Team in analyzing and organizing the wealth of data accumulated; Mr. R. Spencer, the MSFC C.O.R. for the Study, in planning and directing the overall effort; and Mr. G. Wouch and Dr. D. Ulrich, of GE's Space Sciences Laboratory, in providing supporting space processing data.

As noted in the Phase I Final Report, publication of this Phase II report neither implies NASA endorsement of any specific product, process or experiment identified during this phase of the Study, nor a NASA commitment to pursue any program defined as part of this Study.

INTRODUCTION

This Final Report on Phase II of the Study for Identification of Beneficial Uses of Space consists of two volumes, Volume I - Executive Summary, and Volume II - Technical Report. In the interests of reasonable handling, Volume II has been subdivided into two books. Book 1 is the compilation of technical information generated during the Study.

Book 2 of Volume II is a collection of reports issued in support of analyses of specific approaches generated during the Study.

These reports are included in the Final Report so as to provide interested parties with background information utilized by the Study Team in generating the results given in the preceding portions of the Final Report.

CONTENTS OF VOLUME II, BOOK 2

INTRODUCTION

APPENDICES

- A. Levitation Heating and Melting of Tungsten, G. Wouch ✓
- B. Free Suspension Processing of Oxides to Form Amorphous
Oxide Materials - G. Wouch ✓
- C. Crystals for Surface Wave Acoustic Substrates - D. Ulrich ✓
- D. Space Manufacturing of Surface Acoustic Wave Devices -
G. Sardella ✓

APPENDIX A

LEVITATION HEATING AND MELTING OF TUNGSTEN

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APPENDIX A

LEVITATION HEATING AND MELTING OF TUNGSTEN

BY

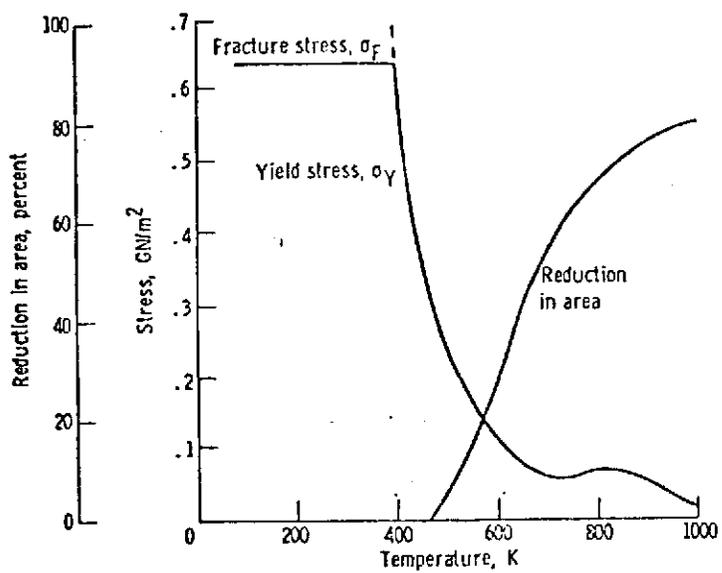
GERALD WOUCH

I. INTRODUCTION

The G.E. Medical Systems Division would like to produce high purity tungsten targets for x-ray production. Tungsten has a high atomic number (atomic number 74) and a high melting temperature (3410°C) capable of withstanding up to 10^7 watts per square centimeter power input levels. The problem is, however, to produce a target having not only good high temperature strength but having ductility at and above room temperature. Solid disc tungsten targets are unusable at the present time because they can be catastrophically fractured in a very few exposures at power levels of about 1×10^5 watts per square centimeter. The present state of the art is an expensive tungsten/10% rhenium layer bonded to a molybdenum substrate. Ductile tungsten would provide a much more fabricable material and be much less expensive to produce.

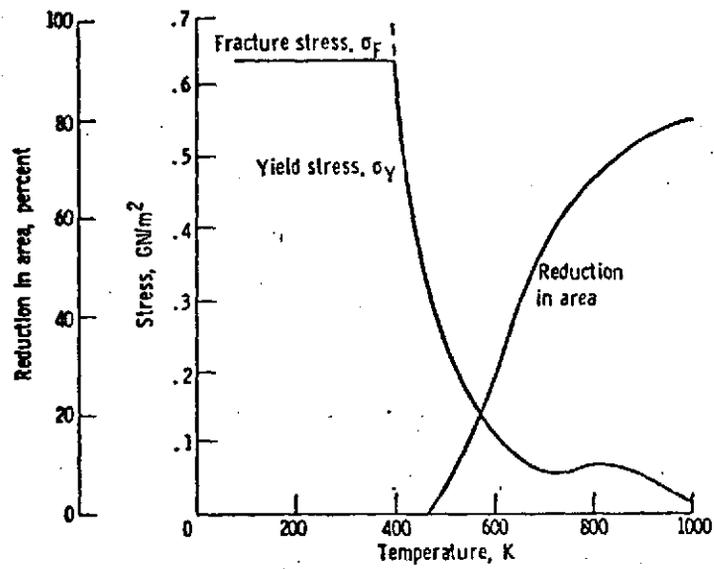
II. BACKGROUND

The problems in producing tungsten with room temperature ductility are illustrated in Figure 1. The material used (Reference 1) was produced by powder-metallurgy techniques and had a recrystallized grain size of approximately 0.29 millimeters in diameter. The yield stress increases sharply as the temperature decreases from 600°K and at about 400°K the yield stress exceeds the fracture stress. The ductility as measured by per cent area reduction falls sharply with temperature and is essentially zero at 475°K . The brittle fracture stress is essentially independent of temperature



-Effect of temperature on flow and fracture characteristics of tungsten. Powder-metallurgy tungsten (200 ppm C; 80 ppm N).

FIGURE 1



-Effect of temperature on flow and fracture characteristics of tungsten. Powder-metallurgy tungsten (200 ppm C; 80 ppm N).

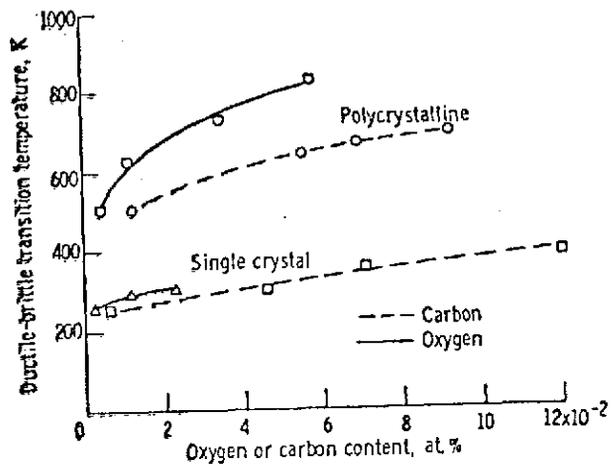
FIGURE 1

below the ductile to brittle transformation temperature (DBTT). Below the DBTT, failure occurred by intercrystalline fracture. This behavior is typical of tungsten produced by powder metallurgy techniques.

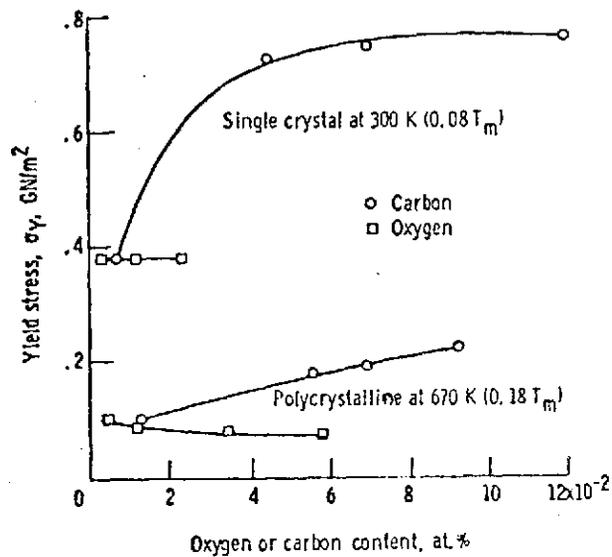
The pertinent factors considered are the effects of interstitial impurities, grain size, and surface condition. Data from many experiments indicate that intergrannular fracture of tungsten is due to the segregation of impurities to the grain boundaries causing embrittlement. Figure 2 shows the effects of carbon and oxygen on the DBTT and yield strength of polycrystalline and single crystal tungsten. It can be seen from Figure 2 that the effect of oxygen and carbon on the DBTT of polycrystalline tungsten is very dramatic while the effect on the yield stress is most pronounced for single crystal tungsten.

Figure 3 shows the effect of grain size on the DBTT and yield strength of tungsten. It is evident from these graphs that very small grains of the order of 0.01 millimeters in diameter favor room temperature ductility and low yield stress. There appears also (Ref. 1) to be a direct correlation between fracture strength and surface features of tungsten specimens. Surface cracks, adsorbed layers and surface grain size may play a leading role in propagating grain boundary fracture, where fracture is propagated from the surface on into the material.

Through controlling the interstitial impurity content, the grain size, and surface features the anticipated product is tungsten with a DBTT at room temperature (293°K) with a yield stress considerably below the fracture stress at room temperature so that tungsten x-ray targets may be fabricated at considerable reduction in cost due to elimination of rhenium

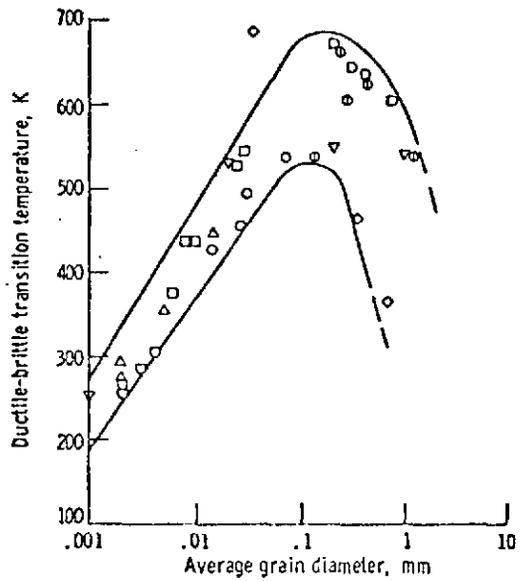


Effects of oxygen and carbon on ductile-brittle transition temperature of single-crystal and polycrystalline tungsten.

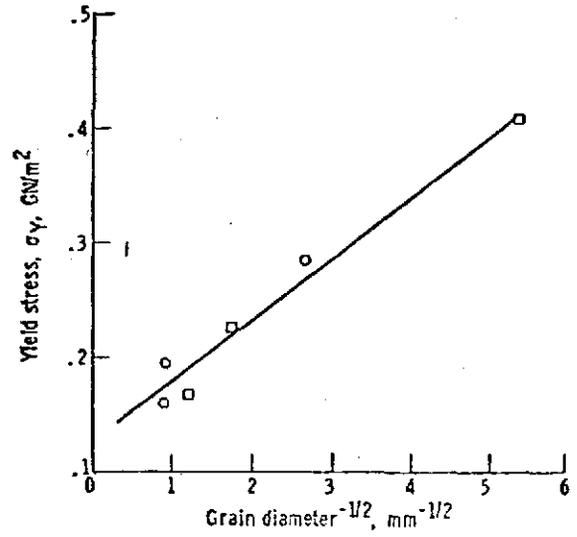


Effects of oxygen and carbon on yield stress of single-crystal and polycrystalline tungsten.

FIGURE 2



- Effect of grain size on the ductile-brittle transition temperature of tungsten.



- Variation of yield stress with grain size at 500 K.

FIGURE 3

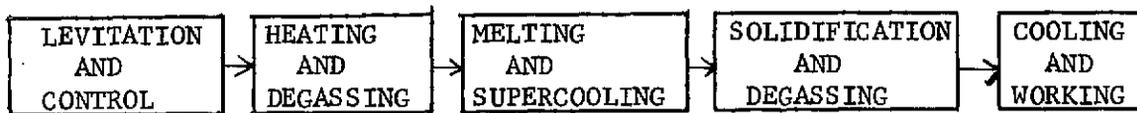


FIGURE 4

and bonding to molybdenum and yet have increased service life from the present 20,000 exposures for the tungsten/10% rhenium bonded to molybdenum targets to a projected 30,000 exposures. The material would have to be produced in pound lots ranging from 0.21 pounds to 2 pounds for the anticipated applications.

III. TECHNOLOGY INVOLVED

Tungsten with room temperature ductility for the manufacture of x-ray targets may be possible through control of interstitial impurity content and grain size. This can be achieved through a process involving the steps of vacuum and/or inert gas purification, melting, supercooling the melt, and initiation of nucleation at the desired nucleation temperature. Figure 4 shows a simplified version of a process line contemplated for levitation heating and melting either in a ground based or weightless facility. The raw material contemplated for this process line is commercial tungsten prepared by powder metallurgical techniques and perhaps recrystallization.

Vacuum and/or inert gas purification in the solid state and the liquid state is employed to significantly reduce the interstitial impurities which cause embrittlement and a high DBTT in tungsten. The pertinent factors to consider in achieving significant purification are:

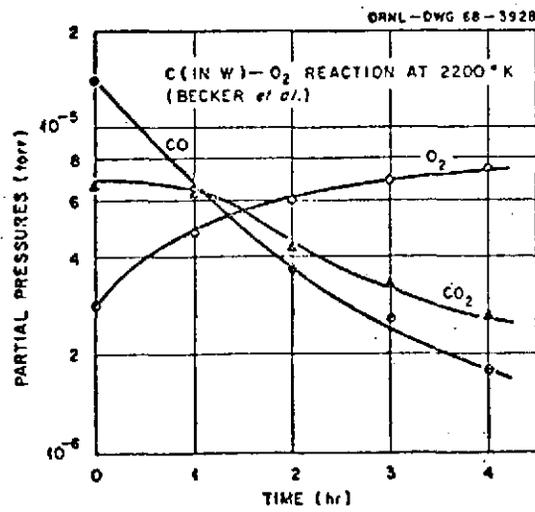
1. the residual gases and their partial pressures in the chamber
2. the temperatures at which degassing is performed
3. the degassing reactions in terms of the reactants, products, chemical equilibria at the degassing temperature and product partial pressures, and reaction kinetics

4. the dwell time at the degassing temperature
5. the vapor pressure of the metal

Solid state degassing of tungsten is generally performed in the temperature range 1800°C to 2400°C with partial pressures of residual gases such as O_2 or N_2 in the range of 10^{-5} to 10^{-10} torr. A prolonged dwell ranging from 30 minutes to several hours at the degassing temperature may be required for significant reduction in impurity content.

Decarburization of tungsten may be taken as a specific example. Figure 5 (Ref. 2) shows the decrease in the partial pressures of CO and CO_2 with degassing time at 2200°K (1927°C) even though the partial pressure of O_2 increases with degassing time. With sufficient oxygen present the decarburization reactions $2\text{C} + \text{O}_2 \longrightarrow 2\text{CO}$ and $\text{C} + \text{O}_2 \longrightarrow \text{CO}_2$ are favored and the reaction rate is governed by the diffusion rate of carbon in the tungsten. The gases evolved CO and CO_2 are pumped away by the vacuum pumping system so that as the carbon content falls due to decarburization the rate of evolution of these gases falls and the partial pressures decrease.

Figure 6 is an illustration in how the solubility of gases in metals generally changes upon melting. For this reason the major degassing should be performed by a prolonged dwell at the degassing temperature or a series of prolonged dwells at selected degassing temperatures before melting. Another reason for this is the formation of bubbles in the melt if the metal has not been degassed. Rapid melting of non-degassed metal may also result in explosive eruptions and spattering. Solidification of a melt with high



Pressures of CO₂ and CO as a function of time due to the decarburization of tungsten with oxygen.

FIGURE 5

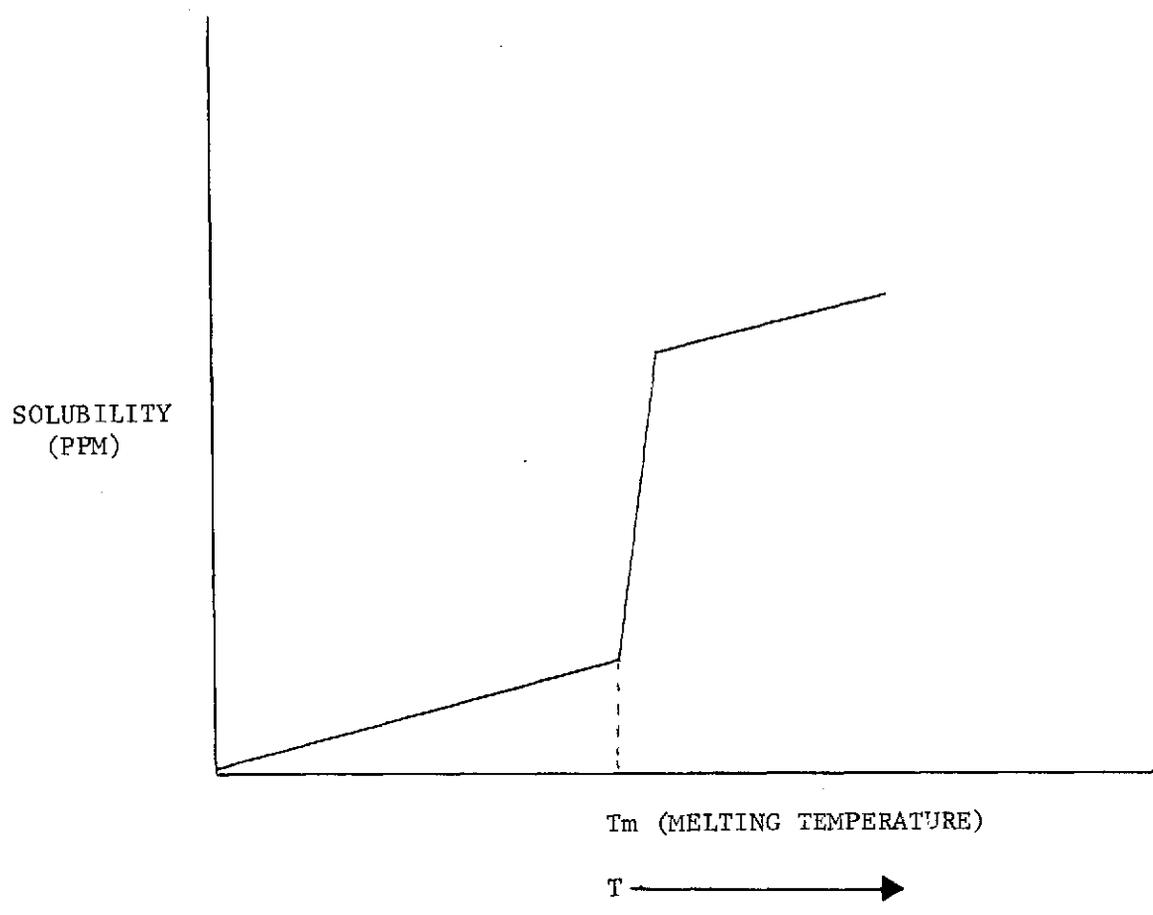


FIGURE 6

gas content and bubbles will lead to porosity and voids due to the change in solubility on solidification, rendering the solid material unusable.

A short dwell after melting the degassed metal with some superheating may result in evaporation of such minor constituents as phosphorous. The pertinent factor to consider is the vapor pressure of the tungsten vs. the vapor pressure of the minor constituent at this temperature. If the tungsten loss is greater than the minor impurity loss, then the impurity content will increase and significant loss of tungsten may also occur.

There are serious drawbacks to inert gas purification. The purity of the inert gas must be extremely high. In one atmosphere pressure of inert gas the partial pressure of active gases should be about 10^{-8} to 10^{-5} ppm. The diffusion of reactants and products through the inert gas is slower so that the incident rate for a given pressure of an active gas at the metal surface is lower in an inert gas than in a vacuum. There are advantages also, however. An inert gas at a pressure of 1 atmosphere will inhibit the formation of bubbles in the molten metal and so help prevent the formation of voids on solidification. It will also slow the evaporation of the molten metal and so prevent losses of tungsten. A combination of vacuum degassing and inert gas atmosphere when molten may provide the best results of purification of tungsten. Gases other than inert gases may also prove beneficial during certain degassing and minor impurity reactions. Hydrogen may be useful in aiding the rapid removal of sulfur and phosphorous and a high partial pressure of oxygen may aid in decarburization.

There are many critical gaps in the vacuum and/or inert gas purification of tungsten which may be filled by ground based research.

These are:

1. dwell temperatures for solid state degassing
2. Environmental Conditions maintained during the process
3. dwell times required
4. dwell time when molten
5. dwell environment when molten.

These gaps may be filled by establishment of a facility to study these problems.

Control of grain size through supercooling molten tungsten may be understood through consideration of Figure 7.

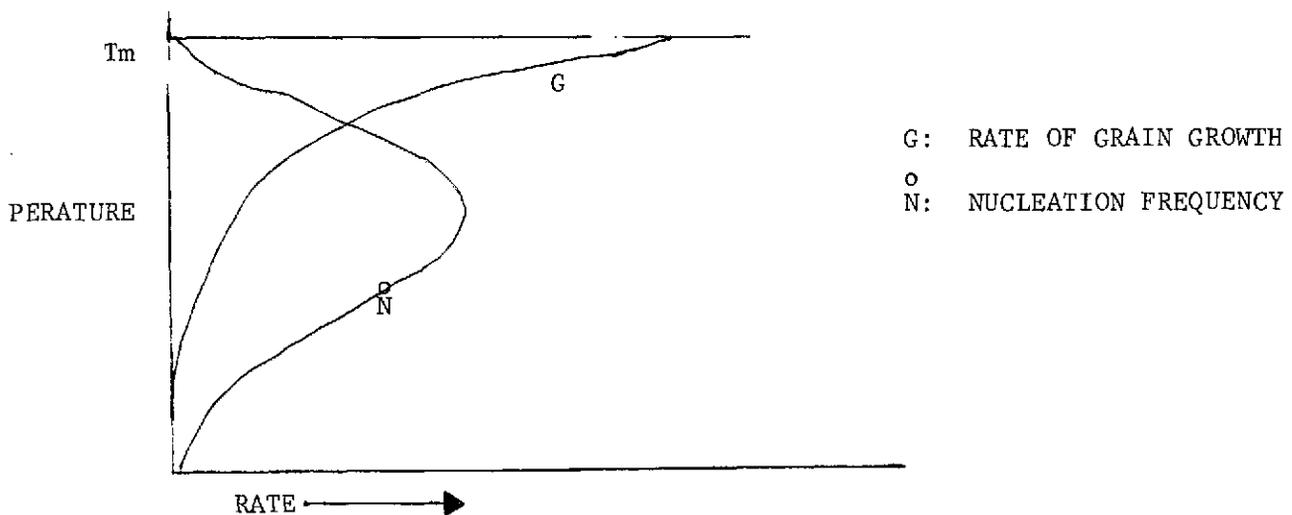


FIGURE 7

Near the melting temperature the nucleation rate is small while the grain growth rate is large so that the microstructure will consist of a few large grains. At the peak of the nucleation rate curve, the grain growth rate is small so that a fine grained polycrystalline structure will result. Thus

if a tungsten melt can be supercooled sufficiently below its melting temperature and nucleation initiated at this temperature, a fine grained polycrystalline material could be obtained. Grain sizes of the order of 0.01 millimeters in diameter would have to be obtained for room temperature ductility according to Figure 3. The achievable temperature to which a high purity liquid metal can be supercooled is usually in the range of $0.82 T_m$ which for tungsten is about 2800°C . Thus 500 to 600°C of supercooling is attainable in a high purity melt if heterogenous nucleation does not occur. Homogenous nucleation is usually inhibited by heterogenous nucleation which occurs on crucible walls and impurity particles in the melt.

There are many critical gaps requiring ground based and/or space experiments for answers. No data exists on the supercooling of tungsten because of its extremely high melting temperature (3410°C). Tungsten can be cast into chilled copper molds but generally due to contamination through crucible melt reactions and heterogenous nucleation at crucible walls significant supercooling is not achievable. Levitation melting and position control either ground based or in 0-g affords a unique opportunity to fill the required critical gaps. These are listed below.

1. Achievable supercooling of molten tungsten
2. Grain Growth and Nucleation Frequency Vs. Temperature
Below the Melting Point
3. The necessity for a rapid gas or liquid quench to retain the microstructure through rapid removal of the released latent heat of melting when nucleation is initiated.

Levitation heating and melting of tungsten offers three unique advantages to the processing of tungsten for x-ray targets. These are:

1. crucibleless heating and melting. Due to the high melting temperature of tungsten (3410°C) there are no crucible materials of high enough melting temperature to contain the melt. Chilled copper crucibles have been used to cast molten tungsten but this has generally not proved satisfactory.
2. vacuum and/or inert gas purification. The degassing of tungsten can be performed without the necessity of a crucible or support which contributes to contamination of the tungsten. Further the levitated piece is more accessible for gas metal reactions as it is suspended in the vacuum or reaction media. When molten, gas bubbles are most often nucleated at the crucible walls. The elimination of the crucible should reduce the danger of gas bubbles which lead to cavities, voids, and porosity after solidification.
3. control of grain size through supercooling. With the absence of crucible walls a very high degree of supercooling of highly purified tungsten may be possible. Eliminating the crucible walls eliminates sites for heterogenous nucleation and after the vacuum and/or inert gas purification process the reduced impurity content will also offer few sites for heterogenous nucleation.

The pertinent factors in levitation heating and melting of tungsten by means of an applied r.f. field are the power absorbed by the material for heating and the force exerted on the material in terms of the material properties. For a single current loop and a sphere of material, the force exerted by the loop on the sphere along the axial line of the loop (Figure 8) is (Ref. 3).

$$(1) \quad F(\text{dynes}) = \frac{3}{50} \pi^2 I_1^2 A(y) G(x) \left(\frac{R_2}{R_1} \right)^3$$

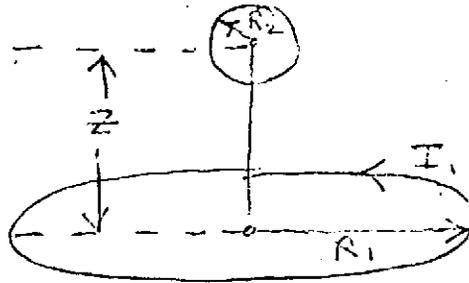


FIGURE 8

where I_1 , is the current in amperes, y is the distance along the axis of the loop in terms of loop radii ($y = z/R_1$) and is a dimensionless quantity, R_1 is the loop radius, R_2 is the sphere radius, x is the ratio of sphere radius to skin depth, δ , ($x = R_2/\delta$). The functions $A(y)$ and $G(x)$ are given by:

$$(2) \quad A(y) = \frac{y}{(1 + y^2)^4}$$

$$(3) \quad G(x) = \left[1 - \frac{3}{4x} \left(\frac{\sinh 2x - \sin^2 x}{\sinh^2 x + \sin^2 x} \right) \right]$$

and the skin depth is given by

$$(4) \quad \delta = \sqrt{\frac{1}{\pi \mu \sigma \nu}}$$

where μ is the permeability, σ the conductivity of the material and ν is the excitation frequency. The dependence with distance enters through $A(y)$ and the dependence with material parameters and excitation frequency through $G(x)$.

The eddy current power absorbed by the sphere from the field is given by (Ref. 3).

$$(5) \quad N(\text{watts}) = 3 \pi R_2^2 \rho_e H^2 F_1(x)$$

where R_2 is the sphere radius in meters, ρ_e the resistivity of the material in ohm meters, H the magnetic field intensity in amperes per meter. Here $F_1(x)$ is given by

$$(6) \quad F_1(x) = \frac{x(\sinh 2x + \sin 2x) - \cosh 2x + \cos 2x}{\cosh 2x - \cos 2x}$$

Figure 9 shows a plot of $F_1(x)$, $G(x)$, and $F_1(x)/G(x)$ versus the material parameter, x . It can be seen from these plots that it is desirable to work above the knee of the $G(x)$ curve which means $x > 5$.

The power radiated by a 2 centimeter molten sphere of tungsten having a mass of 647 grams is given by

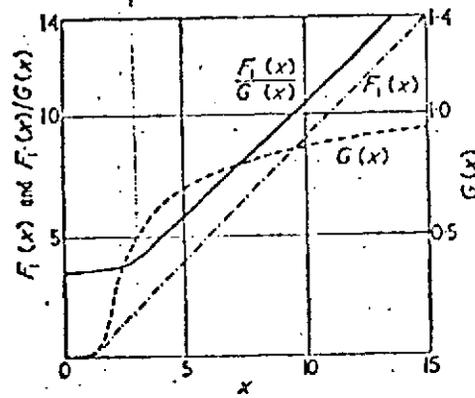
$$(7) \quad N_R = 4 \pi R_2^2 \epsilon \sigma T^4$$

where ϵ is the emissivity, σ is Steffan's constant and T is the temperature in degrees Kelvin. For this size sphere the power radiated is 21,000 watts.

To hold such a sphere molten then at least 21,000 watts of power would have to be supplied and to melt the tungsten power in excess of this would have to be supplied. In contrast a ten gram sphere of about a half centimeter in radius will radiate about 1300 watts which would have to be supplied in order to hold that sphere molten. The power to melt and hold the tungsten molten is the dominant consideration in developing a facility for melting tungsten by any method.

Using the above formulae, to levitate and hold a 2 centimeter sphere of tungsten a coil of at least 6250 ampere turns excited at a frequency of at least 400 kilohertz would be required. With a coupling efficiency of 10% the facility would be at least a 210 kilowatt facility. Because of the large power radiated the power requirements and frequency requirements are identical for both the ground-based and weightless facilities. It is not the requirement for levitation that is important but the power that must be supplied to the sphere to keep it molten. In contrast a 20 kilowatt facility would be adequate for ten grams of tungsten.

Levitation melting utilizing alternative sources of heating power may be very attractive. Such alternative power sources as solar power or electron beam melting with greater efficiencies in coupling heating power into the material are attractive alternative sources. For processing in space this has decided advantages, for only position control is required,



The ratio $F_1(x)/G(x)$ and the functions $F_1(x)$ and $G(x)$.

FIGURE 9

since the ball is freely floating. Thus the power for processing might be considerably reduced through an alternative heating power source.

There are several critical gaps which need to be answered by experiments to assess the feasibility of either ground based or weightless levitation melting. These are:

1. optimum coil shapes and sizes for efficient coupling into the tungsten spheres
2. emissivity of liquid tungsten
3. the possibility of ground based levitation of tungsten without pouring through the coil system.
4. available power for melting in weightless facilities

The expected advantages and improvements through space processing are:

1. the ability to perform vacuum and/or inert gas purification without the necessity of crucibles which introduce contaminants into the tungsten and can nucleate gas bubbles at the crucible melt interface leading to gas cavities, voids and porosity after solidification.
2. the ability to melt and supercool tungsten to temperatures far below the melting temperature. This will enable a very fine grained tungsten to be produced.

3. the final product will be tungsten with room temperature ductility which can be used to produce long exposure life tungsten x-ray targets which presently cannot be manufactured.

Figures 10 and 11 show the preliminary estimation of analysis, laboratory experiments, and space experiments required to establish the feasibility of producing tungsten x-ray targets by either ground based or 0-g processing.

REFERENCE 1: Stephens, Joseph R., "Review of Deformation Behavior of Tungsten at Temperatures Less Than 0.2 Absolute Melting Temperature", NASATMX-2482.

REFERENCE 2: INOUE, H, "Interactions of Refractory Metals with Active Gases In Vacua and Inert Gas Environments", from Refractory Metal Alloys, Metallurgy and Technology, Plenum Press, New York, 1968, pages 165 to 197.

REFERENCE 3: FROM, E. and JEHN, H., "Electromagnetic Forces and Power Absorption in Levitation Melting", British Journal of Applied Physics, 1965, Vol. 16.

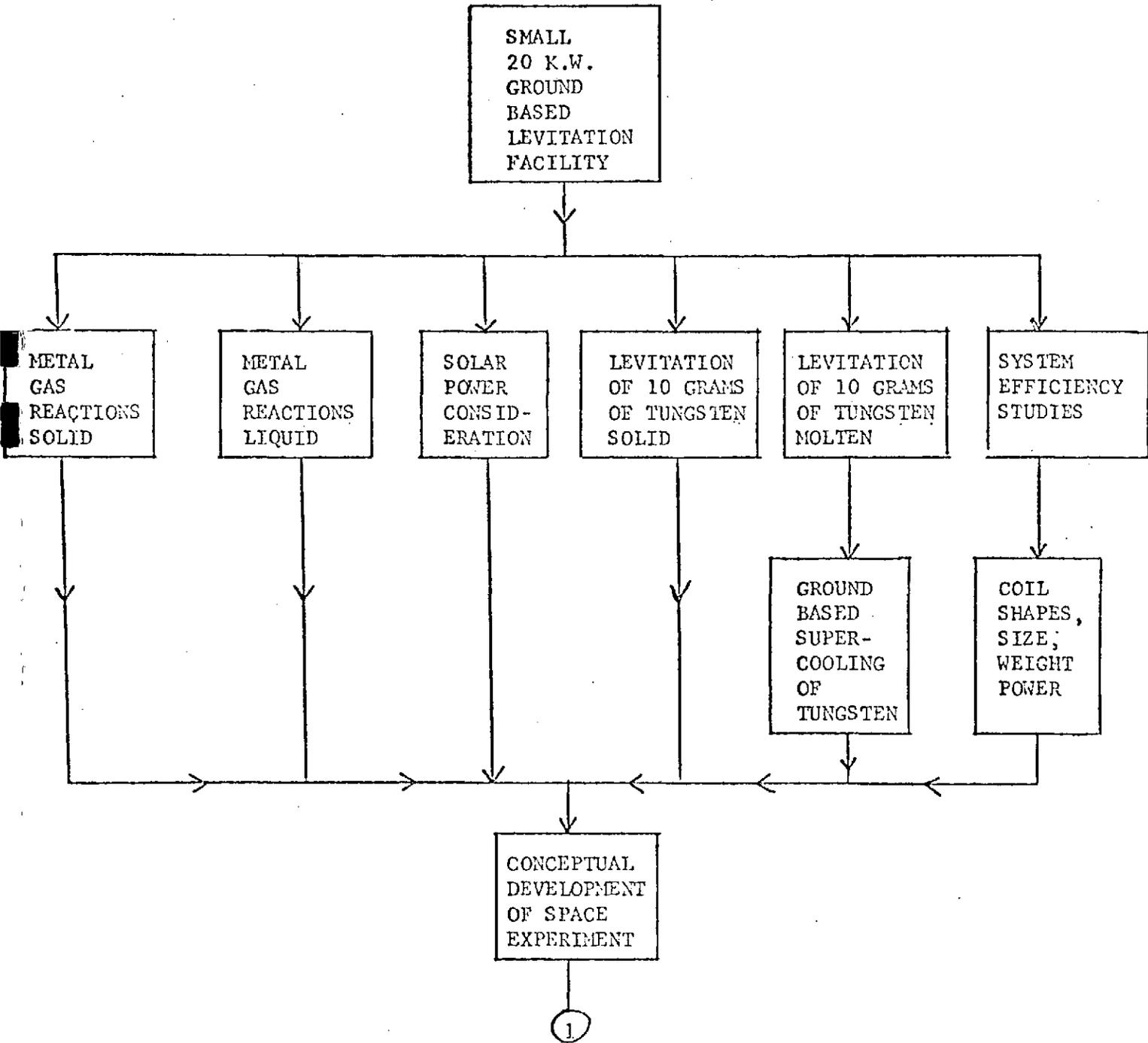


FIGURE 10

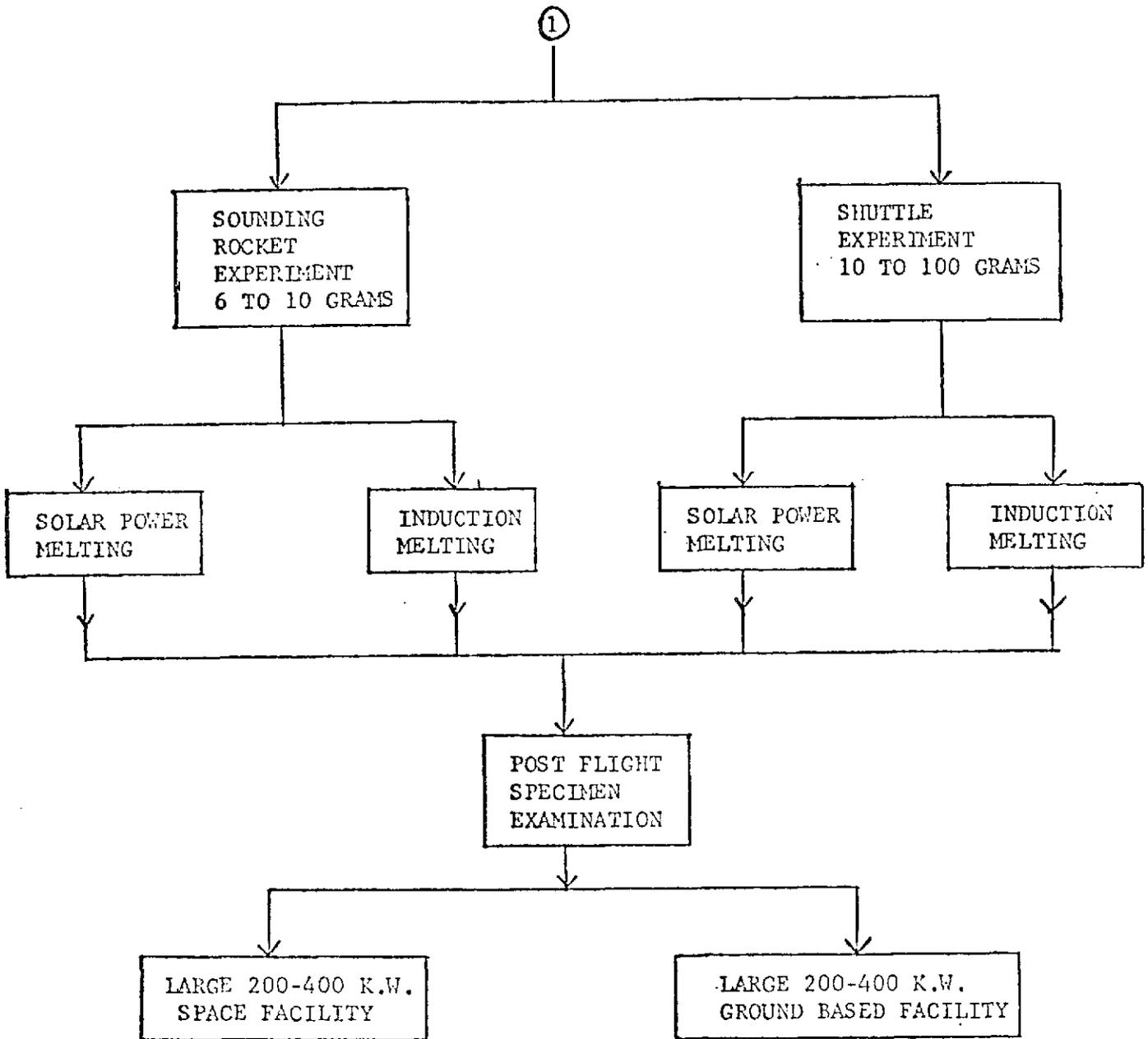


FIGURE 11

APPENDIX B

FREE SUSPENSION PROCESSING OF OXIDES
TO FORM AMORPHOUS OXIDE MATERIALS

June 29, 1973

N74-33401

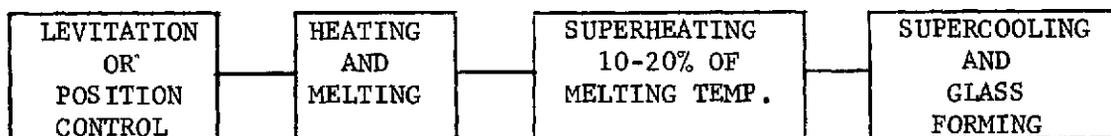
APPENDIX B

FREE SUSPENSION PROCESSING OF OXIDES
TO FORM AMORPHOUS OXIDE MATERIALS

BY G. WOUCH

I. INTRODUCTION

The processing of oxides such as yttria, zirconia, and alumina to produce amorphous transparent oxides may involve a process line such as that shown below.



It does not appear likely that the use of high vacuum conditions will benefit this process because of:

1. Evaporation and decomposition of the compounds used;
2. Plating of the chamber and cooler surfaces;
3. Bubble formation due to outgassing products such as oxygen;

This does not preclude the melting of these materials at vacuum levels suitable for the use of such heating candidates as the electron beam however. The prevention of evaporation of volatile constituents and the decomposition of the compounds used requires a careful consideration of gas-liquid phase equilibria and reaction kinetics. It may be possible to adjust the partial

pressures of the product species so that degassing reactions and sublimation of volatile constituents will not be favored and so operate under conditions of low or medium vacuum, thus favoring certain melting techniques such as the electron beam. This situation requires careful consideration to fill in the knowledge gaps.

The strongest candidates for levitation in a one gravity environment or position control in the weightless environment appear, at this time, to be acoustic, gas stream, and electrostatic. This is because of the high electrical resistivity of alumina, zirconia, and yttria, which are, at best (zirconia) above 10^{-3} ohm meters at temperatures as high as 2000°C and may be (alumina) as high as 10^{20} ohm-meters at room temperature.

Electromagnetic positioning should not be overlooked, however, as present research and development in this area has been directed toward good conductors. For poorer conductors such techniques as confinement in a microwave cavity should be investigated. It may be that a system analogous to confinement in acoustic cavities can be constructed with highly competitive features.

The strongest candidates for heating are hot chamber walls (a furnace), focussed radiation from a source such as the sun (solar heating) or an arc, electron beam heating, laser heating, and possibly, some form of microwave heating. Induction heating of alumina, zirconia, and yttria does not appear favorable. However, heating by indirect methods such as induction heating of a susceptor which would heat a non conductor by radiation from its hot surfaces should not be overlooked either. Each heating technique must be examined in the light of specific environmental conditions such as chamber pressure, residual gases present, and material

properties.

II. LEVITATION AND POSITION CONTROL

A. Acoustic Levitation

The principle of acoustic levitation and acoustic position control is well understood. A sheet of material such as glass or steel will experience a pressure on it when placed in the way of an intense sound beam. This excess pressure is called the radiation pressure. The radiation pressure can be calculated from the sound pressure by the relation

$$(1) \quad P_R = (1 + \gamma) \frac{P^2}{\rho C^2},$$

where P_R is the radiation pressure, P is the sound pressure, ρ is the density of the gas, C the velocity of sound in the gas, and γ the ratio of the two specific heats of the gas. The radiation pressure is normally quite small. For example a sound pressure of 1000 microbars gives a radiation pressure of about 1.8 microbars (1 microbar = 1 dyne/cm²). This corresponds to a sound pressure level ($20 \log P/0.002$) of about 133 decibels which is not untypical in sonic booms. To obtain radiation pressures of 1000 dynes/cm², then 3.75×10^4 milibars or nearly 160 decibel sound pressure levels would have to be employed. To achieve ground based sonic levitation of materials, then, sound level pressures up to as much as 200 decibels would have to be employed. These sound pressure levels are well above human tolerance and are characterized as macro noise. A ground based sonic levitation system would have to be well shielded to prevent hazardous exposure to extreme sound levels. It is important to realize that these sound pressure levels are those required inside the

levitation chamber. The techniques exist for adequately shielding or damping out hazardous noise radiation so that personnel will not be affected when operating such a levitation system. The Jet Propulsion Laboratories have demonstrated a levitation chamber for radiation pressures of about 2 microbars, which requires 133 decibel sound pressure levels inside the chamber by the above calculations. Outside the chamber, however, the sound pressure levels were below 75 decibels. It is expected that much better reduction in sound pressure levels can be obtained so that this problem of noise radiation may not represent a serious problem for acoustic levitation system development. It is one of the problems that must be considered, however, when levitation system design is considered.

For acoustic position control in the weightless environment accelerations as high as 10^{-2} gravities or about 10 cm/sec.^2 would have to be controlled. For a hundred gram mass this would correspond to a 1000 dynes of force. Considering a density of about 5 gms/cm^3 (typical of alumina, zirconia, and yttria) 100 gms corresponds to a cube of about 20 cm^3 volume or about 2.72 cm edge length. The area of a face is about 7.4 cm^2 and a radiation pressure of 135 milibars would be required to exert the force of 1000 dynes on the cube face. Using (1) this requires about 9×10^3 milibars of sound pressure or about 153 decibels sound pressure level. Against accelerations of 10^{-3} gravities 13.5 milibars of radiation pressure would be required or 2.9×10^3 milibars of sound pressure, corresponding to a sound pressure level of about 143 decibels. Thus even in the weightless environment of space dangerously high sound pressure levels inside the levitation chamber must be employed for position control of appreciable amounts of material. The problem of shielding such a facility must be

carefully considered but does not represent a serious design problem.

Acoustic levitation and position control has some distinct advantages which are pertinent to alumina, yttria, and zirconia. The major advantage is that it is not limited to levitation of good conductors. It does not introduce contaminants into melts as an inert gas can be used as the medium for energy transport. It can provide turbulence for mixing if this is required. Acoustic levitation has been successfully demonstrated using rectangular cavities. A standing wave pattern is produced in such a way that the radiation pressure will always urge the mass to the chamber center. Such a system has been developed by the Jet Propulsion Laboratories.

The effect of high sound pressure levels on liquids must be carefully considered. In some liquids cavitation will be introduced and the melt will contain gas bubbles, which cannot be tolerated in glass production. This is a critical knowledge gap which must be investigated. High sound pressure levels may initiate undesirable nucleation and prevent formation of amorphous materials. A program of ground based research is required to fill in such critical knowledge gaps.

B. Electromagnetic Positioning

Electromagnetic levitation and positioning has mainly been applied to good conductors such as the metals. With poorer conductors, such as the oxides, however, electromagnetic positioning is a strong competitor, if higher frequencies than have been considered are considered. This leads naturally into consideration of microwave frequencies and cavity resonators. The conceptual system would basically be similar to acoustic levitation insofar as a standing wave pattern in the cavity would confine

the oxide material in the cavity and prevent the molten oxide from touching the cavity walls.

Electromagnetic positioning forces can be exerted either through eddy current induction by a time harmonic magnetic field or through electric polarization induced by a time harmonic electric field. It is this latter mechanism which has generally not been considered. The force exerted by a time harmonic magnetic field, produced by a current loop, acting on a sphere of radius R, is given by

$$(2) \quad \bar{F} = \frac{2 \pi R^3 G(X)}{\mu_0} (\bar{B} \cdot \nabla) \bar{B}$$

where \bar{F} is the vector force, \bar{B} is the magnetic field, X is the ratio of sphere radius to skin depth, and G(X) is a function given by

$$(3) \quad G(X) = 1 - \frac{3}{4X} \left[\frac{\sinh 2X - \sin 2X}{\sinh^2 X + \sin^2 X} \right]$$

The skin depth is given by

$$(4) \quad \delta = \frac{1}{\sqrt{\pi f \sigma \mu}}$$

where f is the frequency, σ the conductivity, and μ the permeability of the material of which the sphere is composed. The force exerted by a time harmonic electric field on a material sphere, similarly, is given by

(5) $(\bar{P} \cdot \nabla)\bar{E}$ where \bar{P} is the induced polarization and \bar{E} is the electric field. For an isotropic, homogenous dielectric, the polarization induced by a time harmonic electric field $\bar{E} = E \cos \omega t \hat{k}$ is

$$(6) \quad \bar{P} = 4\pi \epsilon_0 R^3 \left[\frac{N_e - 1}{N_e + 2} \right] \hat{k}$$

where ϵ_0 is the dielectric constant of free space (8.854×10^{-12} farad/meter) and N_e is the relative dielectric constant of the material. Thus

$$(7) \quad \bar{F} = 4\pi \epsilon_0 R^3 \left[\frac{N_e - 1}{N_e + 2} \right] E \frac{\partial E}{\partial z} \hat{k}$$

for a time harmonic electric field $E \cos \omega t \hat{k}$.

With a time harmonic magnetic field, the parameter X, is very important and this is highly depending on the electrical conductivity. As an example, alumina at room temperature has a resistivity of 10^{20} ohm meters or a conductivity of 10^{-20} mho/meter. Since

$$(8) \quad X = \frac{R}{\delta} = \sqrt{\pi f \sigma \mu} R, \text{ at a frequency of } 10^{10} \text{ hertz}$$

(X-band radar), a 1 centimeter sphere of alumina would have

$$X = 2\pi \sqrt{10^{-17}} \cdot 10^{-2} = 2\pi \cdot 10 \cdot 10^{-11} \approx 2 \times 10^{-10}$$

For this value of X, G(X) is effectively zero and the positioning force is zero. Zirconia at room temperature, however has a resistivity less than 1 ohm meter and at 10^{10} hertz, a 1 centimeter sphere has an X of

$$X = 2 \sqrt{10^3} \cdot 10^{-2} = (2 \pi) (31.6) 10^{-2} \approx 2.$$

The value of $G(X)$ here is about 0.1 which is still very small. However, consideration of (2) will show that accelerations of the order of 10^{-3} gravities can be controlled. Thus for zirconia, electromagnetic positioning through eddy current induction could be possible, while for alumina it is not possible.

Consideration of time harmonic electric fields however is encouraging. Alumina, zirconia, and yttria have a relative dielectric constant of 5 or better. Considering (7), then, for a one centimeter radius sphere

$$\bar{F} = 6.35 \times 10^{-7} E \frac{\partial E}{\partial Z} \hat{k}$$

For $E = 10^5$ volts/meter and, with $\frac{\partial E}{\partial Z} \approx \frac{2 \pi}{\lambda} E = \frac{2 \pi}{3} 10^{10} E$ for a 10^{10} hertz wave.

$$\bar{F} = \frac{12.70 \pi}{3} 10^{-5} \hat{k} \text{ newtons} = \frac{(12.7) \pi}{3} \hat{k} \text{ dynes,}$$

so a force of about 13 dynes can be produced on all three oxide metals with the time harmonic electric field. This would be sufficient to control 10^{-4} g accelerations for a 20 gram mass of oxide. Going up in field to 5×10^5 volts/meter would increase the acceleration control by a factor of 25 so that accelerations of the order of 3×10^{-3} g could be controlled for a 20 gram (about 1 cm radius) sphere of these oxides.

Thus it appears that a microwave cavity resonator at L band radar frequencies operating at 5×10^5 volts/meter could control accelerations of 10^{-3} g for 20 grams of material. Since the breakdown voltage of air at 20° C is about 3×10^6 volts/meter, this is a factor of 6 below the air breakdown voltage. The breakdown voltage for the gas environment of the molten oxides may limit the position control capabilities of the cavity resonator. This is a critical knowledge gap, which must be investigated. It appears, though, that electromagnetic positioning using microwave hardware is still a strong competitor for the oxide materials.

C. Electrostatic Levitation and Positioning

The use of electrostatic forces for levitation and position control requires careful consideration. A very well known and important theorem of electrostatics (Earnshaw's Theorem) states that no charge can be in stable equilibrium in an electrostatic field under the influence of electrical forces alone. This theorem very generally states that a static system consisting of particles which either repel or attract one another with forces varying inversely with the square of their distance is not stable and that small displacements lead to non-equilibrium configurations. This does not preclude electrostatic levitation and positioning, however. What it means is that an electrostatic levitation or positioning system must be an active system, capable of adjusting charge in such a way as to maintain the specimen at the desired position. There is no potential well the specimen can settle into and remain there as in electromagnetic levitation and positioning.

Two systems which can be considered are

1. Utilization of an inhomogenous electric field to induce an electric dipole moment in the specimen.
2. Creation of a charge on the floating specimen upon which the electric field can act.

For the first system the force exerted on the specimen is given by:

$$(2) \quad \vec{F} = (\vec{P} \cdot \nabla) \vec{E}$$

WHERE \vec{F} is the vector force, \vec{P} is the induced dipole moment, ∇ is the Laplacian operator, and \vec{E} is the electric field. For the second system the force is simply given by

$$(3) \quad \vec{F} = q \vec{E}$$

where q is the charge on the body.

Both the electric polarizability and the charge q will depend on the temperature of the floating mass and this factor must be considered.

The major problems to be encountered with electrostatic positioning systems are:

1. arcing or gaseous discharges
2. controlling the charge on the specimen as the temperature changes.

These require consideration of electrode sizes and shapes, electrode spacing, specimen size, total pressure within the chamber, the residual gases present and their partial pressures, the volatile constituents subliming from the specimen and their vapor pressures, the gases evolving from the specimen and their partial pressures, and the total charge on the specimen. The Earth Orbital Applications Group of the General Electric Space Sciences Laboratories has made some preliminary studies on electrostatic levitation and positioning. A design concept has been formulated which would lead to a system capable of controlling accelerations as high as 10^{-3} g for 10 gram specimens in low, medium, or even high vacuua. Using an inert gas with near atmospheric pressures presents a problem however from the standpoint of charge regulation, which is attained by using an electron beam. If the amorphous oxides can be produced in low or medium vacuua with adjusted gas partial pressures to prevent sublimation and degassing, then such a system would be feasible.

Electrostatic levitation and position control systems are, as yet, in the conceptual stage. Because of this it is difficult to assess what size masses can be controlled and what powers are required. This is a critical knowledge gap which must be filled by experiment.

D. Gas Stream Position Control

Gas stream position control can mean either position control through the use of gas jets or position control through control of the movement of the gas within the processing chamber. The latter could be accomplished through the use of fan blades or positions to create gentle gas streams which would move the material to the desired position. Such a position control system

is potentially a very strong competitor as it would be applicable to either conductors, semiconductors, or insulators and would probably have very low power requirements for controlling 10^{-3} g acceleration levels. It is also clear that this is a gentle method of positioning, which would not appreciably distort or excite oscillations in a molten mass, provided the momentum of the gas stream is kept sufficiently low.

III. HEATING AND MELTING

A. Thermal Imaging Furnaces

Thermal imaging furnaces heat the work piece by focussing upon it radiant energy from a source such as the sun or an arc. Thermal imaging furnaces include:

1. Solar Furnaces
2. Carbon Arc Imaging Furnace
3. Mercury Vapor Arc Imaging Furnace
4. Xenon Vapor Arc Imaging Furnace
5. Tungsten Vapor Arc Imaging Furnace

They have several unique advantages for melting refractory oxides such as alumina, zirconia, and yttria. These are:

1. Attainment of very high temperatures of the order of 4000° K, enabling the oxides considered to not only be melted but superheated.
2. Isolation of the work piece from the heat source thus eliminating disturbances due to electric and magnetic fields, hot, reactive furnace gases, and hot components.
3. Melting of materials in any desired atmosphere or in low, medium, or high vacuum.
4. Melting semiconductors and insulators without susceptors or other heated components, thus enabling a freely floating specimen to be heated and melted.
5. The furnaces are easy to control, their heating pattern is reproducible and relatively stable, and they are of moderate size and cost.

To utilize images as a technique for melting refractory oxides the following considerations are required:

1. the source
2. the source temperature
3. the source geometry
4. the source spectral and geometric emissivity
5. the focussing system with its reflectivities and optical characteristics
6. the receiver of the radiation, its geometry, spectral absorbance, and its conduction, convection, and radiation losses.

The earliest thermal imaging furnaces were solar furnaces. Imaging of very high temperature sources dates back to Archimedes who reportedly burned the Roman fleet besieging Syracuse with mirrors arrayed in a large hexagonal group. In more recent times solar furnaces have been used to melt small specimens of such high melting materials as tungsten.

The features of the solar source are:

1. Equivalent blackbody temperature of 6000°K
2. Angle subtended by the sun 0.00931 radians.
3. On the earth imaging the sun is dependent on weather and the atmosphere through which the radiation must pass.
4. The sun can be treated as a disc source.
5. The radiation received varies from the center of the sun to the edges as $(\cos)^{1/3}$ or $(\cos)^{1/4}$.
6. At most 45% of the energy incident at the top of the atmosphere reaches the focusing system.

The small angle subtended by the solar source limits drastically the spot size on the work piece and the diameter of this is given by $D=0.00931 f$,

where f is the focal length of the parabolic mirror usually employed in the solar furnace. The 34-inch focal length of the 120-inch Convair-Conn solar furnace has an image diameter at the vertical of about 0.34 inches. Other high temperature sources can have larger images and are available at will and readily controlled. A small solar furnace comprising a fixed parabolic mirror 2m in diameter and 0.85 meters in focal length captures approximately 3m^2 of radiation normal to the sun (about 3 kilowatts). The useful dimensions of the image is 13 to 14 mm in diameter with a mean illumination of 900 W/cm^2 and of 1500 W/cm^2 at the center. The efficiency as a measure of the solar radiation captured is 40%. The same mirror in space would capture about 6KW of energy and give a mean illumination of 1800 W/cm^2 and about 3000 W/cm^2 at the center. To melt large specimens of high melting temperature materials such as alumina, yttria, and zirconia using the solar source parabolic reflectors up to 10 meters in diameter with focal lengths up to 600 centimeters would have to be employed. Such mirrors have been made as a composite of mirror plates. Spot sizes of 55.8 centimeters can be obtained. A large semi-industrial unit such as this has been built in France. An output of 132 lb. per hour of steel in continuous operation was obtained (weather permitting). While this is low for steel output, for a high cost specialty item such as the transparent amorphous oxide melts, this may be a reasonable output. With the 10 meter diameter composite mirror used in France about 60 kilograms per day of fused or fritted zirconium oxide (zirconia) has been produced. This installation gathers about 50 kilowatts of solar energy.

From the above, it is clear that mirrors up to 2 meters in diameter are adequate on Earth or in space for heating, melting, and supercooling experiments. The obvious advantage is that whatever the efficiency, the power gathered is free so that power sources are not required. For small-scale

specialty production of such materials as transparent amorphous oxides solar mirrors up to 10 meters in diameter would be required. These would be composite mirrors constructed out of small sections which can easily be transported on the shuttle and assembled during extra vehicular activity. Before shuttle descent, the mirror would be disassembled and stowed for use on other shuttle flights.

The Carbon-Arc Imaging furnace has been used by Kestigian et al (Ref. 1) to prepare single crystals of titania, alumina, magnesia, zirconia, and thoria. Temperatures in the range of 3300 C can be reached with colorless compounds. The furnace of Kestigian et al is described in Ref. 1. It uses two carbon sources, each with a lifetime of 20 minutes apiece so that a total of 40 minutes of lifetime can be obtained without having to pause to put in new sources. With sequential operation of the sources an indefinite operating period can be obtained. Over a horizontal profile of 0.8 cm the irradiance varies from about 200 cal/cm² sec at the focal point to about 120 cal/cm² sec at the edges. Pressed pellets 3/8 in. thick by 3/8 in. diameter of titania, alumina, magnesia, zirconia, and thoria were melted, cooled, and remelted.

Carbon arc and other arc imaging furnaces appear to be a useful tool for investigating space processing of transparent amorphous oxide materials. Their major disadvantages are high input power requirements (perhaps as high as 20 kilowatts) and small spot size. Small spot size does not appear to be a disadvantage for small amounts of material such as 3/8 inch by 3/8 inch buttons of refractory oxides. However, for larger scale work there is a critical knowledge gap in developing larger areas of irradiance. It might be possible, for example, to focus several images on the larger work piece

so as to heat and melt it uniformly.

Of all the thermal imaging techniques, the solar furnace appears best for melting the refractory oxides. The limitation appears to be the largest composite mirror that can be assembled and utilized within the time line of shuttle flights. An interesting experiment would be to place a large composite mirror in orbit with orbit correction capabilities to prevent orbital decay over long time periods and have the shuttle rendezvous with the facility. This would provide an essentially free power source for heating and melting large pieces of material.

B. Laser Heating and Melting

Laser heating and melting suffers from many misconceptions. Initially the laser output energies were often described in terms of the number of razor blades which could be penetrated and all sorts of marvelous materials applications were foreseen. Initially, lasers did not have a high average power capability, however. While they could focus enormously high energies into a small spot for short transient periods they could not put the sustained power into a piece required to melt and hold it molten. Thus lasers were, then, categorically eliminated as possible materials processing instruments. With the increase in average power from milliwatts to tens and then hundreds of watts laser welding of thin pieces of material and applications in microelectronics became possible. Now with average powers above a kilowatt and reaching toward 10 kilowatts, laser heating and melting of such high melting temperature materials as alumina, zirconia, yttria has become possible.

The CO₂ laser will probably become the workhorse of industry laser applications as its average power capability is pushed beyond 10 kilowatts (Ref. 2).

The CO₂ laser shows great promise in competing with conventional electron beam units for heating and melting. The efficiency of the CO₂ laser, however, still is less than 20% contrasted to electron beam efficiencies which can be as high as 75%. A physically compact 1.1 kilowatt CO₂ laser, obtaining this power from a 1 meter active medium length at 10% overall efficiency has been described in Ref. 3. It is expected that the higher average power CO₂ lasers (up to 10 kilowatts) can be obtained without too much extension of active medium lengths. This is only the active medium, length, however. Coupled with heat exchanger, blower, and electrical input power system and the device can be quite bulky. With a 10% overall efficiency an 1100 watt CO₂ laser would require 11 kilowatts and a ten kilowatt CO₂ laser would require 100 kilowatts of energy.

The advantages of laser heating is that it can be employed in any desired environment, such as low, medium, or high vacuum or inert gas. With sufficient power the laser could melt any small specimen (≈ 10 grams). For pieces of 100 grams or larger powers up to twenty kilowatts might be required with 10-20% superheating and with only 10% efficiency this means a 200 kilowatt facility.

It is clear that the use of lasers for heating and melting indicates continuous output. To obtain the average powers discussed a pulsed laser putting out short transient pulses would have to put enormous power into the pulse (10^{12} watts/cm²) and this would evaporate rather than melt.

While these pulsed power lasers are desirable for machining and cutting, they are not desirable for heating and melting.

Summarizing this discussion we have the following points.

1. New lasers with up to 10 kilowatts average power will be available.
2. The efficiency of these lasers is 10-20% maximum.
3. The continuous output type could be used to heat and melt samples of alumina, yttria, and zirconia. Small samples up to 10 grams could be melted with the 1.1 to 2 kilowatt CO₂ lasers available now. Larger specimens could be melted with the 10 kilowatt units to be later available. They do require input powers of 100 kilowatts for large pieces (\approx 100 grams to 1 lb.), however.

C. Induction Heating

Direct eddy current induction heating of zirconia, alumina, and yttria does not appear practical because of their poor conductivity. Indirect induction heating is very practical, however. Indirect induction heating consists of induction heating a good conductor surrounding the work piece to high temperatures, which then heat the work piece by radiation from the hot walls. With a tungsten susceptor of correct size and shape for the r.f. coil system, temperatures to 3000 C could be reached. The tungsten susceptor could be fashioned to be a furnace enclosure and an acoustic cavity for simultaneous heating, melting and positioning or even a microwave cavity for high frequency dielectric positioning.

The disadvantages of this technique is the power required to heat the tungsten walls. Power inputs as high as 25 kilowatts would be required even for good coupled susceptor-r.f. coil systems to melt large pieces of alumina, yttria, and zirconia (\approx 100 grams or more).

Even with this disadvantage induction heating by indirect methods seems to be one of the systems to be considered for transparent amorphous oxide production. Small susceptor furnaces could be built for initial experiments with 10 grams of the oxides, incorporating either acoustic or microwave dielectric positioning. Larger production furnaces could be built, then for pilot plant operation. The only limitation would appear to be available power for the furnace.

D. Microwave Heating

Alumina, yttria, and zirconia are relatively lossless dielectrics. Therefore microwave dielectric heating is not practical.

E. Electron Beam Heating

Electron beam heating of zirconia, alumina, and yttria is definitely feasible. These materials have a high enough coefficient of secondary electron emission so that levitated pieces could be melted by beams with 10 K.v accelerating voltage and less than one ampere of beam current. The electron beam melting must be performed in a relatively good vacuum (10^{-2} torr or less) and so this precludes melting in an inert gas. Melting in a good vacuum with adjusted partial pressures of residual gases to prevent degassing and sublimation, however, should be carefully considered. Under such conditions electron beam is not only desirable but it is the most efficient technique available (75% efficiency).

There is a large critical knowledge gap in the materials processing of these oxides which has to be answered in order to assess the applicability of electron beam melting. It is necessary to determine the degassing, sublimation, and dissociation reactions which deteriorate these oxides and to see if vacuum levels compatible with electron beam melting can be maintained while these reactions are controlled.

F. Other Techniques

Other heating techniques include chemical flames, the plasma torch, contact melting, arc melting, and the hot wall furnace. These techniques subject the work piece to contaminants, reactive gases and hot furnace components. It is difficult to maintain a controlled atmosphere with these techniques. In the case of a hot wall furnace, tungsten would seem to be the only suitable material for furnace construction.

G. Summary

<u>Technique</u>	<u>Advantages</u>	<u>Disadvantage</u>	<u>Recommendation</u>
Solar Furnace	<ol style="list-style-type: none"> 1. Free Power 2. Can melt any solid. 3. Melting in any desired environment 4. Heating can be easily controlled 	<ol style="list-style-type: none"> 1. Available power determined by mirror size. 2. Spot size determined by mirror focal length. 3. Requires a special time line for maximum utilization. 	<ol style="list-style-type: none"> 1. Small solar mirrors up to 2 meters for initial exp. 2. Large composite solar mirrors up to 10 meters for specialty production.

<u>Technique</u>	<u>Advantages</u>	<u>Disadvantages</u>	<u>Recommendation</u>
Arc Imaging Furnace	<ol style="list-style-type: none"> 1. Can Melt any solid 2. Melting in any desired environment. 3. Heating can be easily controlled. 	<ol style="list-style-type: none"> 1. Requires high power inputs. 2. Small spot size. 	<ol style="list-style-type: none"> 1. Used for initial experiments on small work pieces only.
Lasers	<ol style="list-style-type: none"> 1. Melting in any desired environment. 2. Heating can be easily controlled. 	<ol style="list-style-type: none"> 1. Low mean power (≈ 2 kw max. now) 2. Small spot size. 	<ol style="list-style-type: none"> 1. Small samples up to 10 grams.
Induction Heating by Indirect Method	<ol style="list-style-type: none"> 1. Can melt alumina, yttria, and zirconia. 2. Melting in any desired environment. 3. Heating can be readily controlled. 	<ol style="list-style-type: none"> 1. Requires high power input. 2. High losses from radiation to furnace surroundings. 3. Temperature limited to about 3100°C. 	<ol style="list-style-type: none"> 1. Used for initial experiments on small work pieces only.
Microwave		<ol style="list-style-type: none"> 1. Will not heat such lossless dielectrics. 	<ol style="list-style-type: none"> 1. Not recommended.
Electron Beam	<ol style="list-style-type: none"> 1. Efficiency high (75%). 2. Will heat large levitated pieces quickly without high charge buildup if the material has good coefficient of secondary electron emission. 3. Heating can be easily controlled. 	<ol style="list-style-type: none"> 1. Requires high power input. 2. Can only be used in vacuum of 10^{-2} torr or less. 3. Limited to materials which will not build up high charge in free floating state. 	<ol style="list-style-type: none"> 1. Used for both initial and final experiments if environment required permits.

It is unfortunate but true that the clear message from these considerations is that the large composite solar mirrors have an overwhelming advantage over any other heating technique. The power is essentially free and the available power and spot size determined only by the mirror size and focal length. The unfortunate aspect is that the construction of large composite mirrors does not fit into the shuttle program and so apparently cannot be utilized. If there is any possible way to modify this situation it should be attempted. Otherwise, for large piece processing considerable input powers up to a hundred kilowatts or more will have to be available.

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APPENDIX C

SURFACE WAVE ACOUSTIC SUBSTRATES

APPENDIX C

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CRYSTALS FOR

SURFACE WAVE ACOUSTIC SUBSTRATES

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Typical single crystal materials which will be used for the future SWA device substrates include lithium niobate (LiNbO_3), lithium tantalate (LiTaO_3), bismuth germanate ($\text{Bi}_{12}\text{GeO}_{20}$), barium sodium niobate ($\text{Ba}_2\text{NaNb}_5\text{O}_{15}$), and spinel (MgAl_2O_4). The growth techniques, process interactions and defects in these crystals have recently been reported in another GE study (Reference 1). The economics of crystal growth of this type of crystal was also studied (Reference 2).

Lithium niobate is grown from the melt using the Czochralski technique. Although boules as large as one inch in diameter and six inches in length can be prepared in this manner, dynamic growth at elevated temperatures introduces chemical imperfections and inhomogeneities. Compositional non-uniformity along the length of the crystal, non-uniform growth regions and compositional differences from crystal to crystal arise from variations in the melt composition, rate of growth, crystallographic axis of pull, etc. Spiral radial strain patterns can be induced at low crystal rotation rates by convection flow alone.

For surface wave acoustic applications, surface perfection is as essential as internal crystal perfection. Growth ridges interfere with the propagation of high frequency surface waves. Because of mechanical

damage and imperfections introduced even during careful lapping and grinding, polished surfaces do not transmit a signal as undistorted as that expected from an equally large natural flat surface.

For high frequency operation, size becomes most important concurrent with surface smoothness. Long crystals of LiNbO_3 which are six to ten inches in length are desired for dispersive delay lines. Currently, long delays cannot be obtained in available sizes of these crystals. In addition, at Gigahertz frequencies the linewidths of surface electrodes deposited on the crystals are in the range of the crystal surface roughness dimensions.

Lithium niobate crystals grown by flux techniques are often found to contain micro-cracks which distort sonic crystals. These are attributed to constitutional supercooling near the seed or trapped or occluded flux.

It is anticipated that lithium-niobate crystals grown in the space environment, with the attendant benefits of near-zero "G" loadings, minimal convection, etc. may show improvements in each of the above problem areas.

Barium sodium niobate ($\text{Ba}_2\text{NaNb}_5\text{O}_{15}$) is grown by the Czochralski technique. Crystal boules which are one inch in length and 5 millimeters in diameter can be grown. However, yields are low; the density of artifacts is high. There are many kinds of defects, cracking, compositional non-uniformity and striations which degrade sonic propagation. The predominant cause of growth striations in barium sodium niobate has been shown to be due to unstable thermal convection. In addition, the

ferroelectric domain structure is sensitive to the distribution of impurities caused by the hydrodynamic flow effects produced in the melt by the rotating crystal.

Lithium tantalate is grown by the Czochralski technique and is in full scale production as the replacement for quartz filters. Large aspect ratio crystals are grown. However, the defect density of these crystals is reported at this point.

Bismuth germanate ($\text{Bi}_{12}\text{GeO}_{20}$) can be grown as boules from which samples up to 20 centimeters in size can be fabricated. It is grown by melt techniques. Surface imperfections are considered to be a problem. Surprisingly, imperfections in these crystals have not been reported in the detail that lithium niobate or barium sodium niobate has.

Spinel has been grown by both the flux and Czochralski technique. Crystal boules with a high degree of perfection are available on a production basis. It is a candidate for producing low attenuation at high frequencies. Growth striations have been observed in chromium doped MgAl_2O_4 .

It should be noted that research is being conducted on the preliminary design of experiments and the design of facilities for the growth of surface wave acoustic crystals in the Shuttle Sortie Lab. This is being conducted by D. R. Ulrich and co-workers under a follow-on of NASA Contract NAS 8-28114.

Work is being directed toward the solution growth of ceramic oxide crystals such as $\text{Bi}_{12}\text{GeO}_{20}$ and lead germanate $\text{Pb}_5\text{Ge}_3\text{O}_{11}$ with emphasis on the definition of gravity-related defects.

Most of the ground based work which has been conducted on the space growth of crystals has been on metals and semiconductors.

In particular, Johnston and Ruff have studied the influence of gravity on tin single crystals; (Reference 3,4). Gates and Witt have investigated iridium antimonide (Reference 5).

Skylab will carry out crystal growth experiments based on metals and semiconductors. It is not expected that the results can be directly translatable to solution grown oxide crystals.

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APPENDIX D

SPACE MANUFACTURING OF SURFACE ACOUSTIC WAVE DEVICES

SPACE MANUFACTURING OF SURFACE ACOUSTIC WAVE DEVICES

N74-33403

BY G. SARDELLA

1.0 INTRODUCTION

As noted in Reference 1, the discussions of Surface Acoustic Wave Electronic Components indicate that vibration is a prime consideration in maintaining resolution during manufacturing phases. Of particular concern is the lithographic mask fabrication process which utilizes a scanning electron beam to fabricate the electrode pattern on the mask. Another process of concern is the use of the mask and soft X-ray exposure to produce contactless lithographic replica patterns of electrodes with sub-micrometer line width resolution.

In the earth-bound laboratory, a massive concrete base with isolation will reduce many of the ambient vibrations within a building. An existing installation now in use is providing large reductions in vibration, but isolation is not effective at extremely low frequencies. These vibrations, therefore, limit the minimum achievable width of the mask finger and ultimately the operating frequency of the transducer. It would be desirable to reduce vibration even below that which is now obtainable on a concrete base, and in addition, extend the lower end of the bandwidth to frequencies approaching DC. If significant reductions in level could be obtained then the improvements in resolution for manufacture would raise the operating frequency of the transducers.

Space manufacturing can provide a natural and unique low-g environment within an orbiting vehicle. This level would exist down to a very low frequency regime. Disturbances would reduce to that of on-board mechanisms and equipment, orbit correction nozzle firing, venting and that equipment required for life support of a crew. It should be noted that even these disturbances are controllable such that their contribution could be made minimal. Fabrication of the masks, and, possibly manufacturing of the surface acoustic wave components, themselves, aboard a space lab would thus avoid the inherent ground vibrations and the frequency limitation imposed by a seismic isolator pad.

2.0 MANUFACTURING VIBRATION REQUIREMENTS

In the mask fabrication phases of X-ray lithography, if an electrode finger is to be less than one micron wide, then, reasonably, it should be required to maintain resolution to 0.1 micron. A minimum criterion of 10:1 will, therefore, be assumed for vibration displacement with respect to the finger width required. Vibration isolation presently available has permitted electron beam spot diameters in the 200 - 2500 Å range. This represents finger widths of less than 2500 Å and vibration input amplitudes at least one order of magnitude less. These results can be obtained on a concrete seismic block installation. Further improvement can be obtained on an air-spring servo-controlled granite block base. An interdigital transducer was thus produced with a finger width of 1500 Å which operates fundamentally at 3.5 Ghz. Exposure times were on the order of two minutes.

In order to improve resolution beyond that just described design goals can be stated as follows:

Vibration amplitude	-	25 - 100 A ^o
Lower frequency limit	-	DC
Mask writing period	-	Up to 40 Hrs.
Transducer operating frequency	-	30 GHz

An environment which would permit these goals to be approached by significant amounts should yield the desired resolution in manufacturing.

3.0 VIBRATION ENVIRONMENT

Vibration levels measured on the concrete floors of laboratory buildings at GE Valley Forge have been found to vary from 50 to 100 micro g in the frequency range of 2.5 to 250 Hz (see Figure 1). This type of measurement is highly dependent on soil type and condition, building construction details, etc. but were repeated at several locations building sites and at different times. The measurements also compare favorably with ground measurements at other locations (references 2, 3, 4, 5). These ground vibrations were effectively isolated by massive seismic block installations supported by air springs, rubber springs, or on a gravel bed. The difference between floor vibrations and vibration on a spring-mounted seismic mass in the Vibration Lab at Valley Forge can be seen by comparing figures 2 and 3. Here, an order of magnitude gain based on overall g (rms) level was achieved on the seismic mass in the range measured. Figure 4 shows the acceleration spectra measured during tests on a gyro stand at Northrup (reference 6). A factor of 220 gain over ground levels was achieved at 10 Hz on the stand.

Any seismic support system, however, has a low frequency cutoff in the 0.5 Hz to 5 Hz range which prevents isolation of the low frequency accelerations. In addition vibration amplitudes are magnified at the support resonances as shown in figure 5. Vibration amplitudes become even more favorable with servo controlled air springs and have, to date, provided the most practical and best available vibration amplitudes in the frequency range above the system resonance.

An investigation of orbital vibrations was made to characterize the orbital accelerations and compare them to that obtainable on the ground. A limited amount of data were available in time for this report but it is apparent that more is available. Much of the data was recorded for purposes other than environmental evaluation, such as to measure the vehicle acceleration during thruster firings. For example, an orbital accelerometer calibration experiment was made using a Bell milli-g accelerometer during an Agena orbit. A report on this experiment listed in reference 7 describes the nature of this experiment to evaluate nozzle firings. The processing of this data was such for the nozzle firings that it would have to be reprocessed for an "environmental" evaluation. Retrieval of these data from the tape recordings and reprocessing should provide acceleration and frequency information in the under 50 Hz range. On another program, during a Titan/Centaur proof flight, milli-g accelerometers recorded nozzle firings and venting during zero-g coast phase and also during "settled" coast periods (where six pounds of longitudinal thrust firings were made). Although not specifically recorded for this purpose the non-firing periods can be studied and the levels approximated. Figure 6 is a sample oscillogram trace of the subject data. Recorder

sensitivity was such that levels could only be estimated to be less than 20 micro-g. This indicates the acceleration levels are in a range that would provide desirable displacement magnitudes. For example, if we assume the maximum 20 micro-g level at 7.5 Hz which is indicated from the oscillogram we would obtain the following:

acceleration .000020g at 7.5 Hz

$$\begin{aligned} \text{then } y_0 &= \frac{4 \pi^2 a (386)}{4 \pi^2 f^2} \\ &= \frac{0.000020 (386)}{4 \pi^2 (7.5)^2} = 3.48 \times 10^{-6} \text{ in.} = 883 \text{ \AA} \\ &\hspace{15em} \text{(Double Amplitude)} \end{aligned}$$

Due to the system noise levels and recorder sensitivity it is not possible to determine how much lower than 883 \AA the levels at 7.5 Hz are. The significance here is that not only are the orbital acceleration levels less than 20 micro-g but there is no isolator resonance with which to contend.

It is felt that more data can be located and if processed for analysis in the desired frequency range, lower accelerations at frequencies approaching DC could be identified.

4.0 SPACE MANUFACTURING CONCEPTS

Several orbital lab manufacturing concepts have been proposed in the literature which would be applicable for the surface acoustic wave (S.A.W.) transducers (references 8, 9). These would be useable in the Sortie Lab of the Space Shuttle (see Figure 7). Reference 8 describes three approaches, two of which are appropriate:

Serpentuator-Based Experiment

Detached Module-Based Experiment

Either of these modes would provide the low-g environments, without a low-frequency isolator disturbance, with the detached module providing perhaps the most favorable levels. A third approach would be the obvious attached mode where the processing equipment would tie directly to the Sortie Lab payload section structure. Quoting from reference 8 the proposed systems are as follows:

4.1 Serpentuator-Based - "The facility shown in figure 8 in the attached mode can be designed so that the entire chamber can be detached from the guide rails and picked-up by a manned or remote controlled serpentuator. The serpentuator-based experiment offers three unique capabilities:"

a. "The units can be moved further away from the vehicle essentially eliminating heat exchange between experiment and vehicle wall".

- b. "For processes requiring high vacuum, the experiment can be moved into the vacuum wake of the vehicle where vacuum reaches a level of ⁻¹⁴ 10 mm Hg."

c. "The most attractive potential of the serpentuator is the achievement of a "free-flying" mode in which almost absolute zero-g is obtained except for the always-present earth-gravity gradient across the mass. In the attached mode, the position control force, which prevents the material from free drifting on its own trajectory, exerts an often undesirable g-level. The elimination of this force is particularly desirable in fundamental experiments calling for the "cleanest" environmental condition."

"One means of achieving a free-drifting condition is through the use of a detached module whose attitude is controlled by the movement of the test material. Since for most experiments the drifting distance after initial positioning is well within the movement capabilities of the serpentuator, the same effect can be achieved at a substantially lower effort by monitoring the serpentuator position by the drifting test material so it remains in the center of the chamber. The use of the serpentuator for this semi-detached mode is illustrated in Figure 9. The procedure is as follows:"

- a. Deployment of the chamber to extravehicular position.
- b. Activation of position control system.
- c. Pick-up by the serpentuator
- d. Material deployment
- e. Simultaneous de-activation of position control and activation of serpentuator attitude control.
- f. Processing of material
- g. Change-over from serpentuator attitude control to position control.
- h. Return of chamber to deployment apparatus and return to vehicle.

"Position control of the serpentuator may be continuous if serpentuator vibrations can be fully attenuated by a closed-loop servo system. If vibrations cannot be eliminated, position adjustment is carried out in increments, consisting of momentary position sensing, followed by a period of serpentuator movement and vibration damping."

4.2 Detached Module-Based Experiment - "If the free-drifting condition should be maintained over long time periods during which the displacements with regard to the base vehicle are large, a detached module, whose position is controlled by the experiment material is necessary. A potential design for a manufacturing module with detachable sections is shown in Figure 12. It consists of a larger section which remains attached to the base vehicle. After preparation of the experiment, the smaller section

is detached and the experiment is monitored by remote control. The details of such an operation shall not be given here and will require further study."

5.0 RECOMMENDED DEVELOPMENT PROGRAM

The use of an orbital facility for fabrication of the S.A.W. components offers an extremely attractive and realistic opportunity to reach the previously stated goals. The concepts need further study and analysis but the possibilities are apparent. These observations strongly indicate the need for a development program to confirm the potential offered by the use of an orbiting spacecraft. It could be accomplished in steps such that each increment would logically call for or lead to the next. The sequence would be as follows:

- a. Orbital Acceleration Environmental Survey - Complete a survey of existing data on Agena, Centaur and other vehicles. Reprocess tape data and other measurements with required frequency range, sensitivity, etc. to permit the environmental assessment. Reduce all the available data to the common denominator of a spectrum analysis for more accurate comparisons and analysis. Generate the requirements for specific, necessary flight measurements.

- b. In-Flight Environmental Survey - Conduct in-flight vibration measurements on various satellites and the Space Shuttle while in zero-g coast phase. This should be done to meet the requirements of frequency and amplitude established in part a. Measurements should be made with

and without equipment running and any other appropriate orbital perturbation. These acceleration measurements will probably be similar to seismic measurements on the ground thus requiring perhaps a strain gage seismometer, or a milli-g meter. Measurements should be made at potential structural mount interfaces in the Sortie payload compartment.

- c. Conduct In-Flight Experiments - Conduct experiments to determine feasibility, resolution and time periods achievable by one or more of the three possible modes: attached, floating, detached module. Experiment could be a small electron beam mask fabrication setup designed only to verify resolution; or experiments could be a seismic measuring device on a detachable pallet.

- d. Design/Fabricate Test Bed - Perform operational experiments of the Test Bed aboard the Space Shuttle Sortie Lab; determine resolution and other critical S.A.W. component parameters.

- f. Design/Fabricate Permanent Space Facility - Design and fabricate the manufacturing facility. Write operational plan.

- g. Install Facility in Sortie Lab - Operate the space facility.

6.0 CONCLUSIONS

The Space Shuttle Sortie Lab presents an attractive and realistic opportunity to provide an orbiting manufacturing facility for the production of S.A.W. components. Such a facility will greatly enhance the resolution in the manufacturing process and permit the desired improvements in performance. The limited data available at this point not only indicates the environments are attractive but that operations can be practical. It is concluded and recommended that the first steps in the proposed sequence be taken to confirm these observations.

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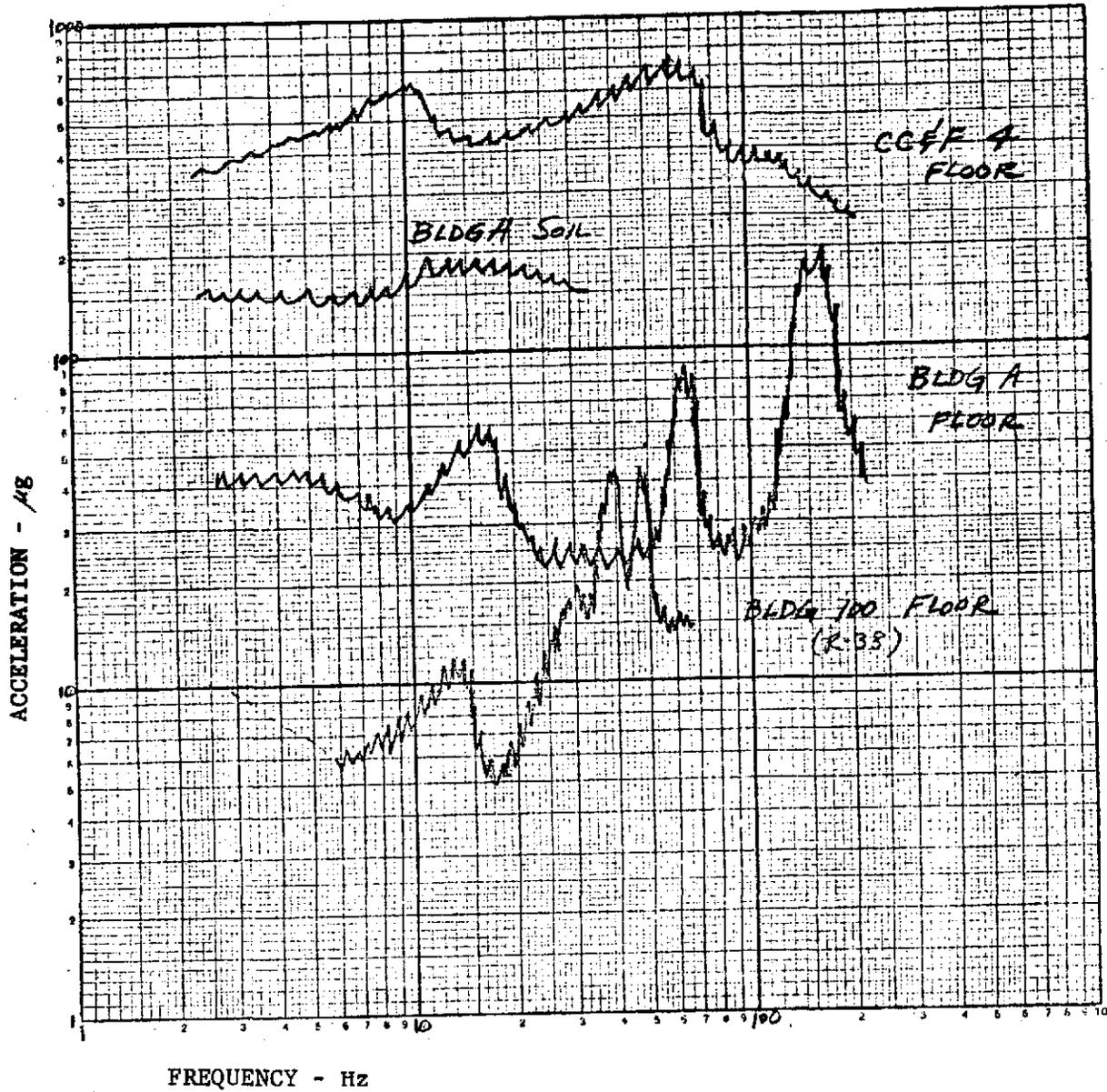


FIGURE 1

VERTICAL GROUND ACCELERATIONS AT VALLEY FORGE UNDER AMBIENT CONDITIONS

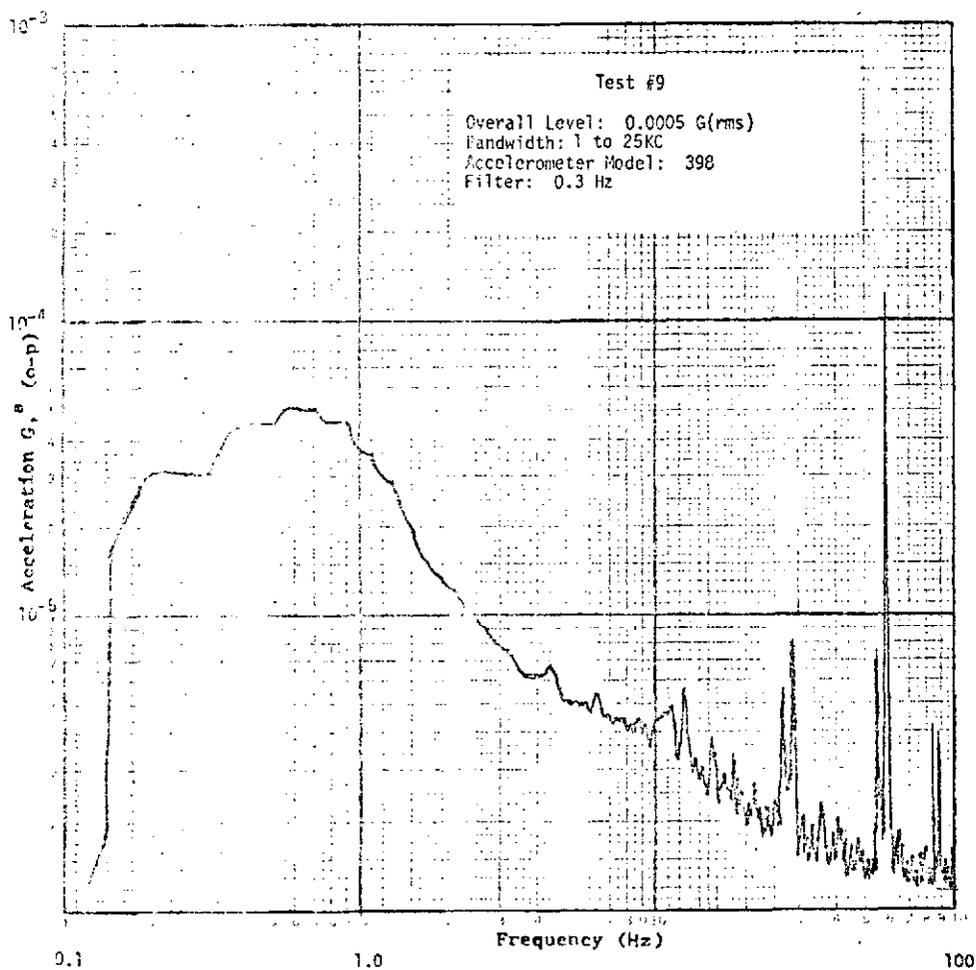


FIGURE 2

SEISMIC MASS VIBRATIONS - V.F. VIBRATION LAB

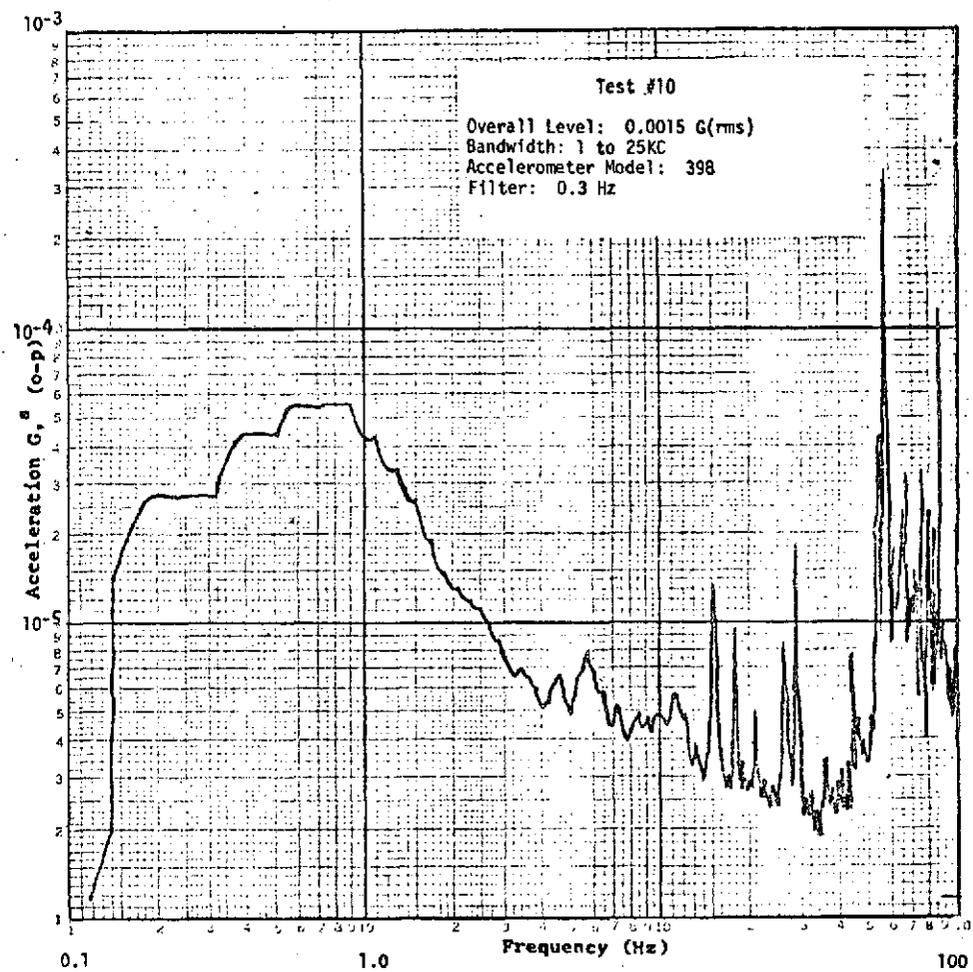


FIGURE 3

FLOOR VIBRATIONS - V.F. VIBRATION LAB

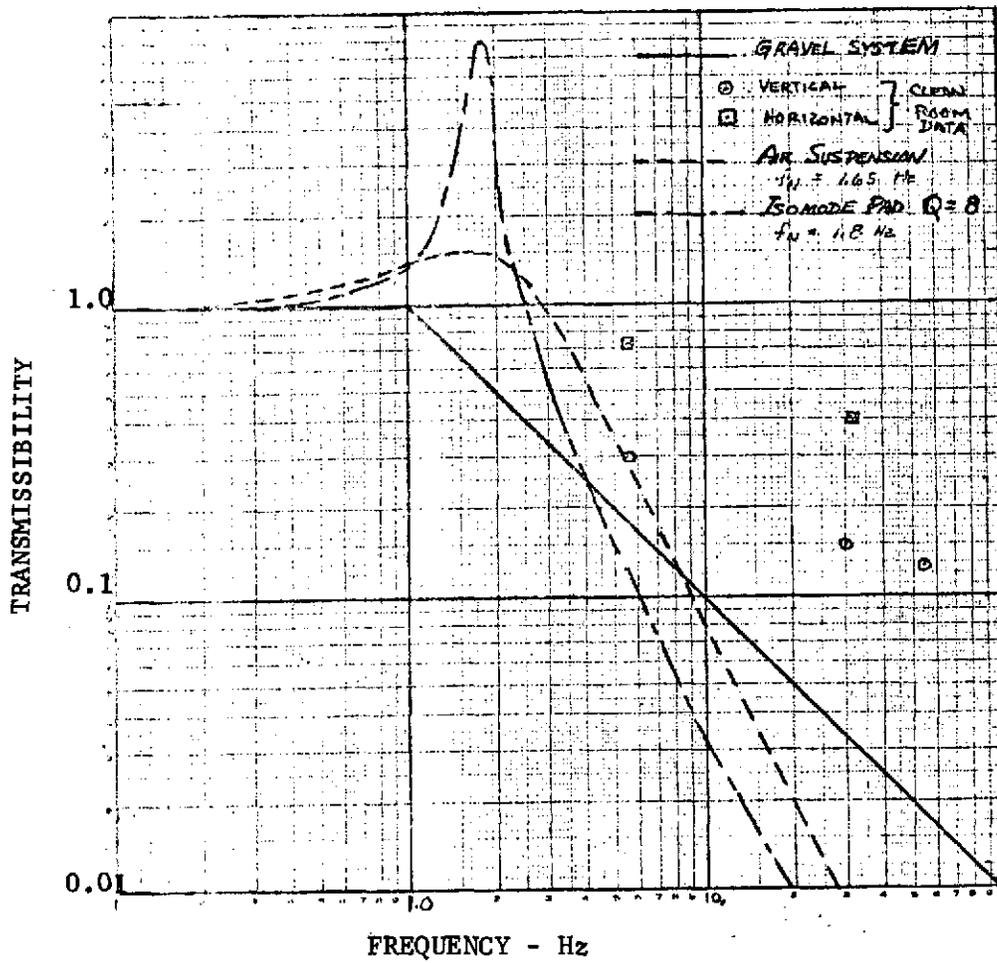


FIGURE 5

VIBRATION TRANSMISSIBILITY OF VARIOUS METHODS
 OF BLOCK SUSPENSIONS

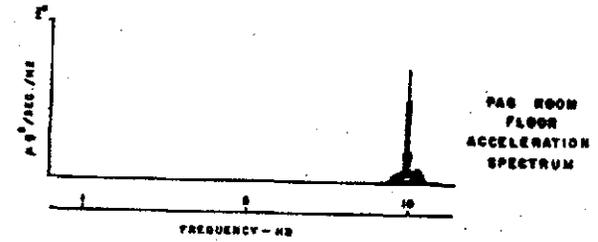


FIGURE 4a Floor Acceleration Spectra

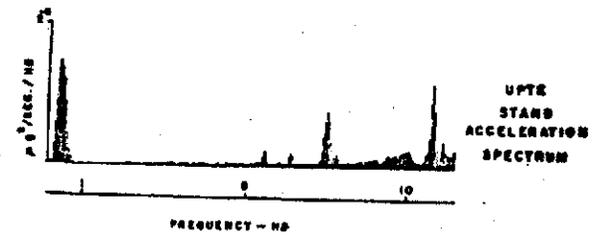


FIGURE 4b Stand Acceleration Spectra

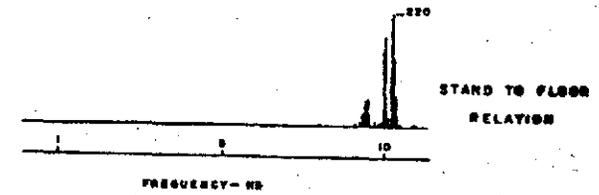


FIGURE 4c Spectral Ratio: Stand/Floor

FIGURE 4

NORTHROP FACILITY VIBRATIONS

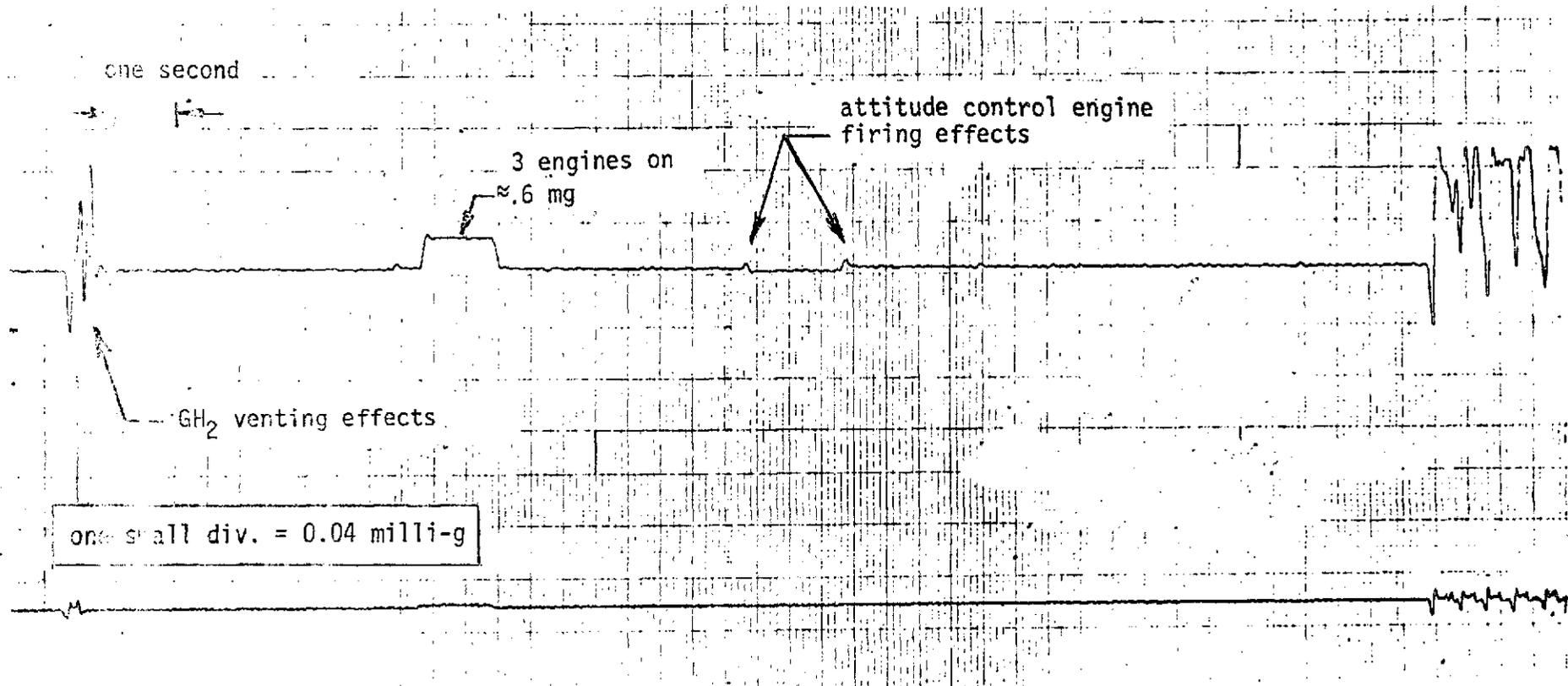


FIGURE 6

CENTAUR ORBITAL ACCELERATION - LONGITUDINAL

SORTIE LAB CONCEPT

PRESSURIZED LAB
UNDEPLOYED
SORTIE LAB SUPPORT
PALLET FOR ALTERNATE OR
GROWTH PAYLOADS

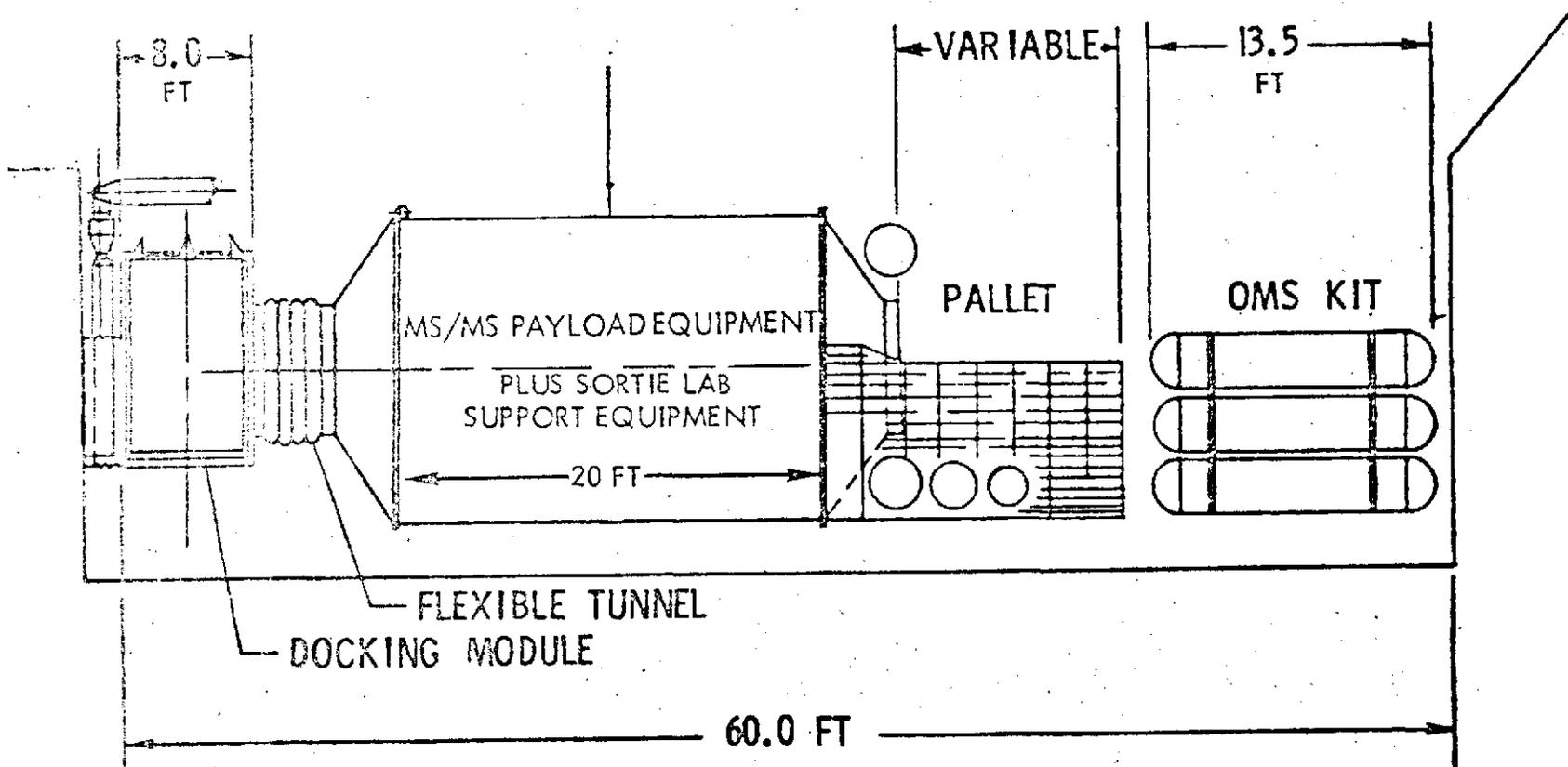


FIGURE 7

SORTIE LAB DESIGN CONCEPT

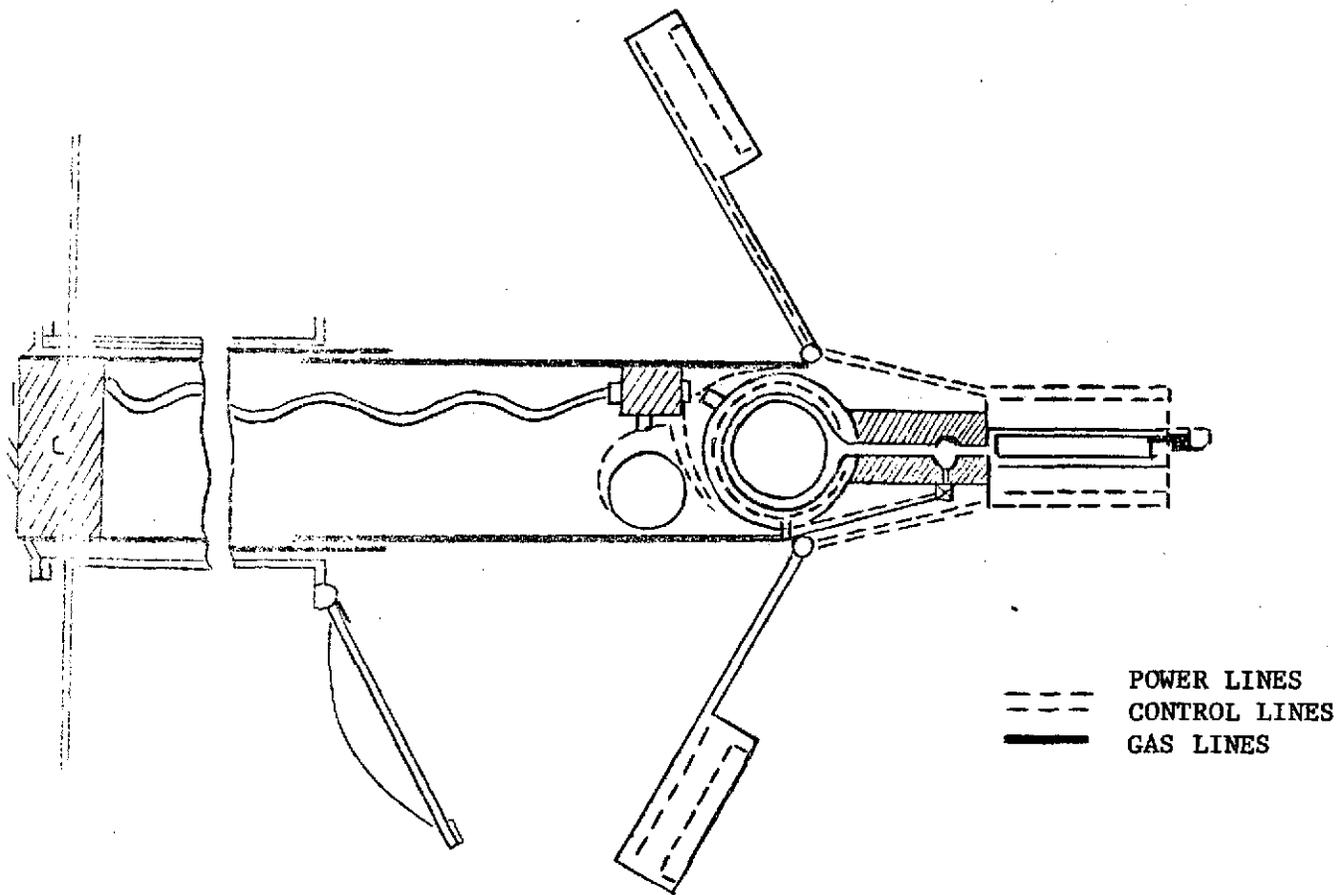


FIGURE 8

ATTACHED MODE, EXTRAVEHICULAR EXPERIMENT

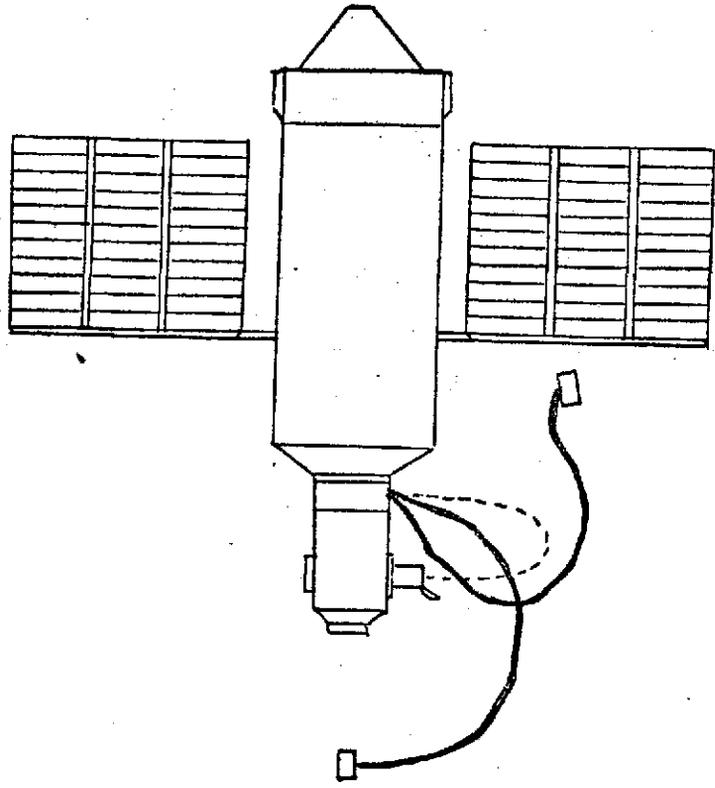


FIGURE 9

SPACE MANUFACTURING OPERATIONS

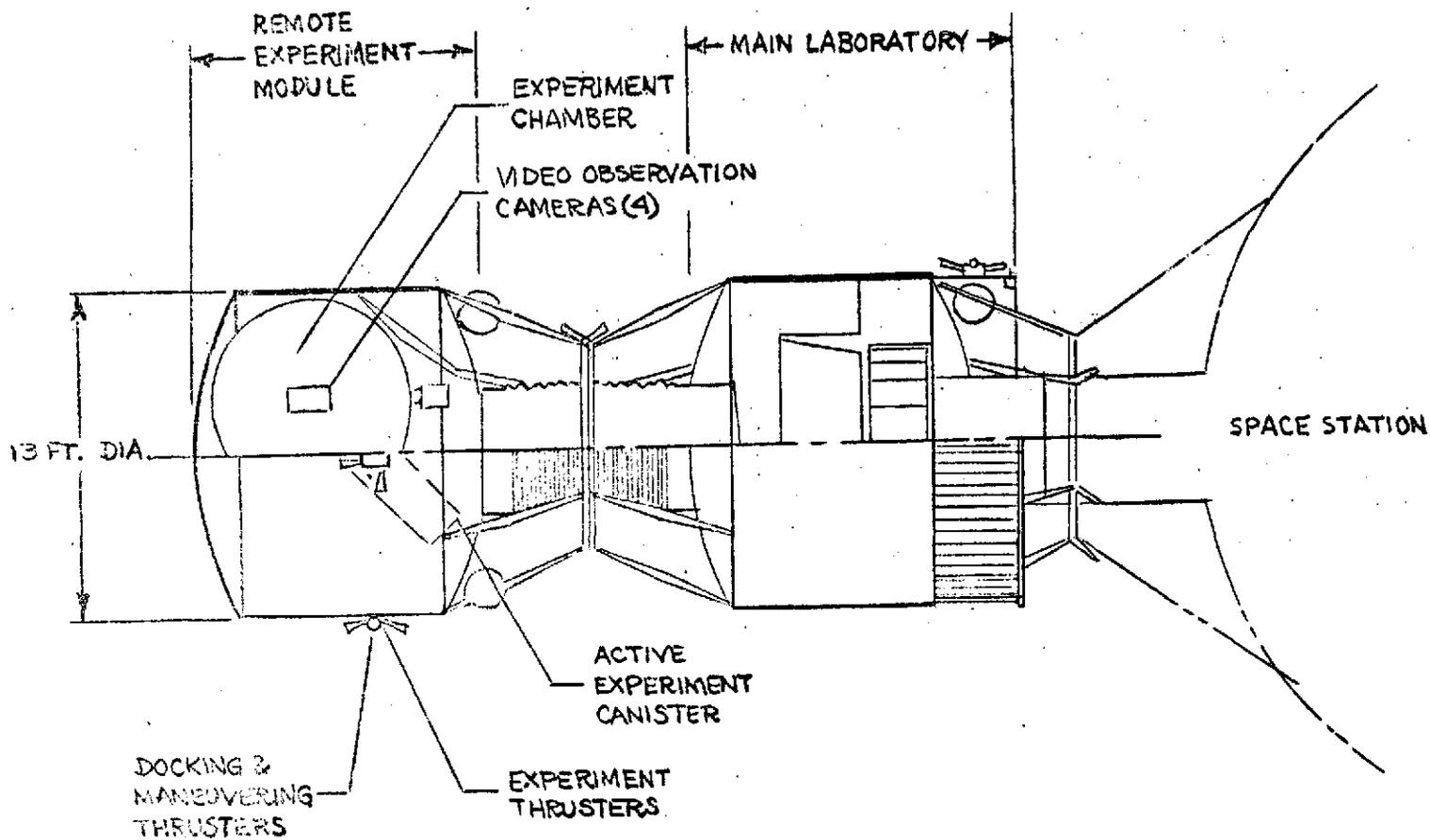


FIGURE 10

MATERIALS SCIENCE AND PROCESSING EXPERIMENT MODULE